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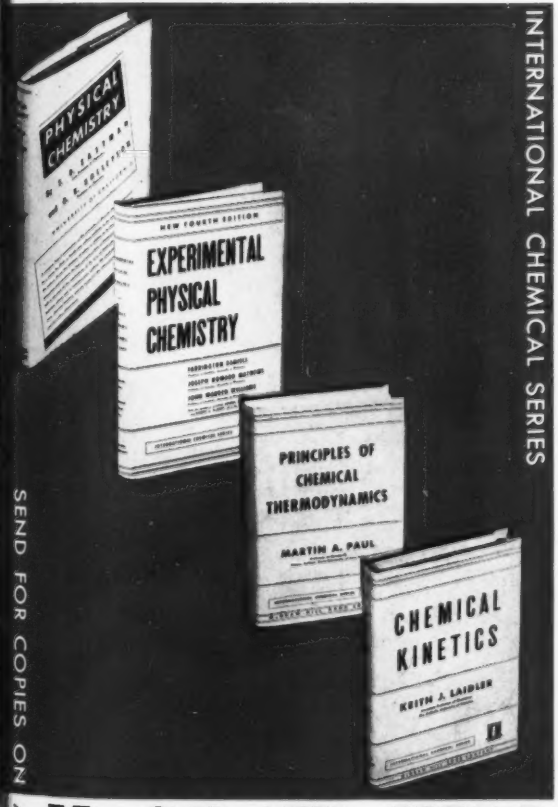

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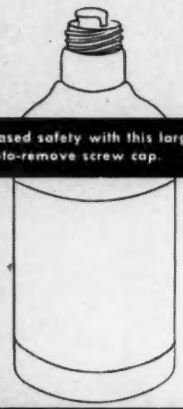

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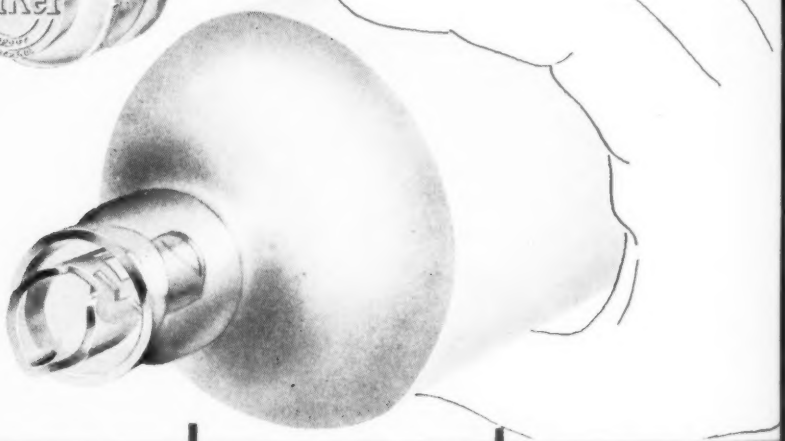

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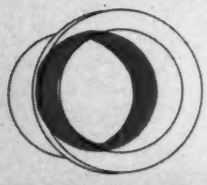

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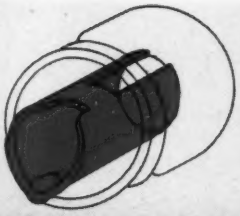

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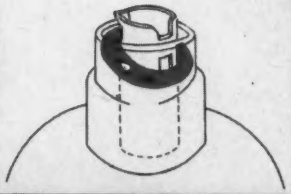

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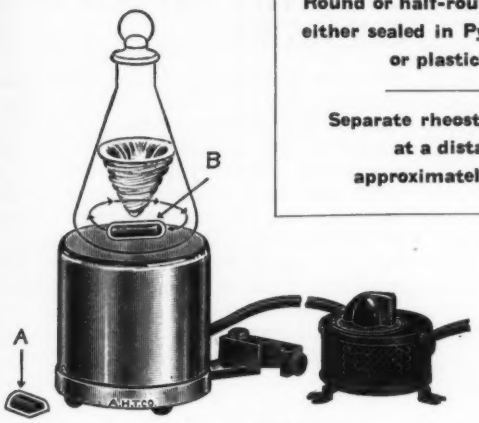

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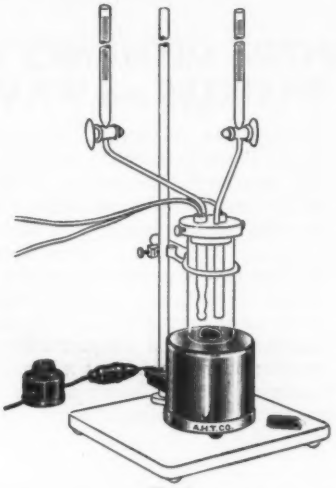

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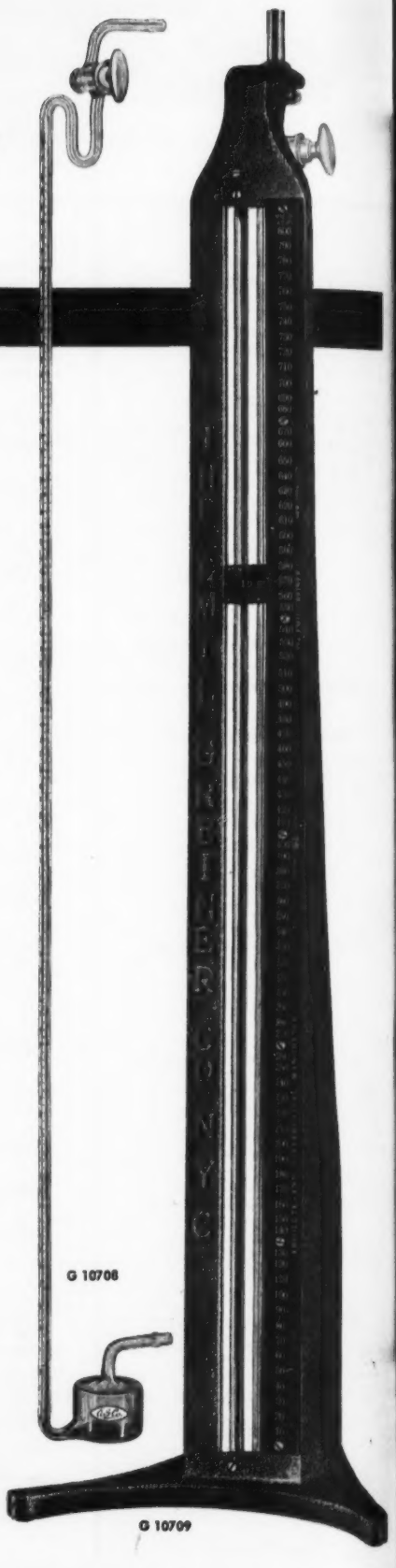

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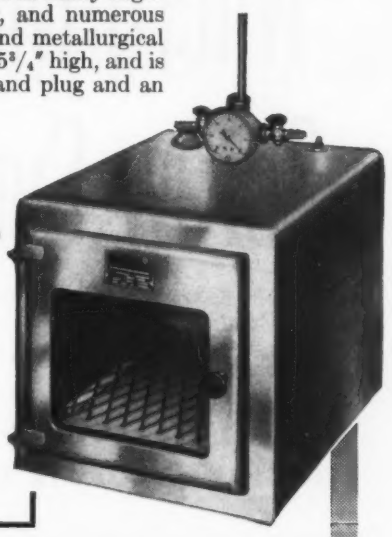

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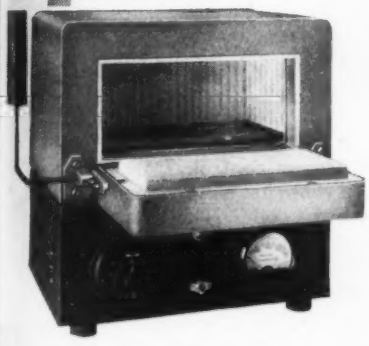

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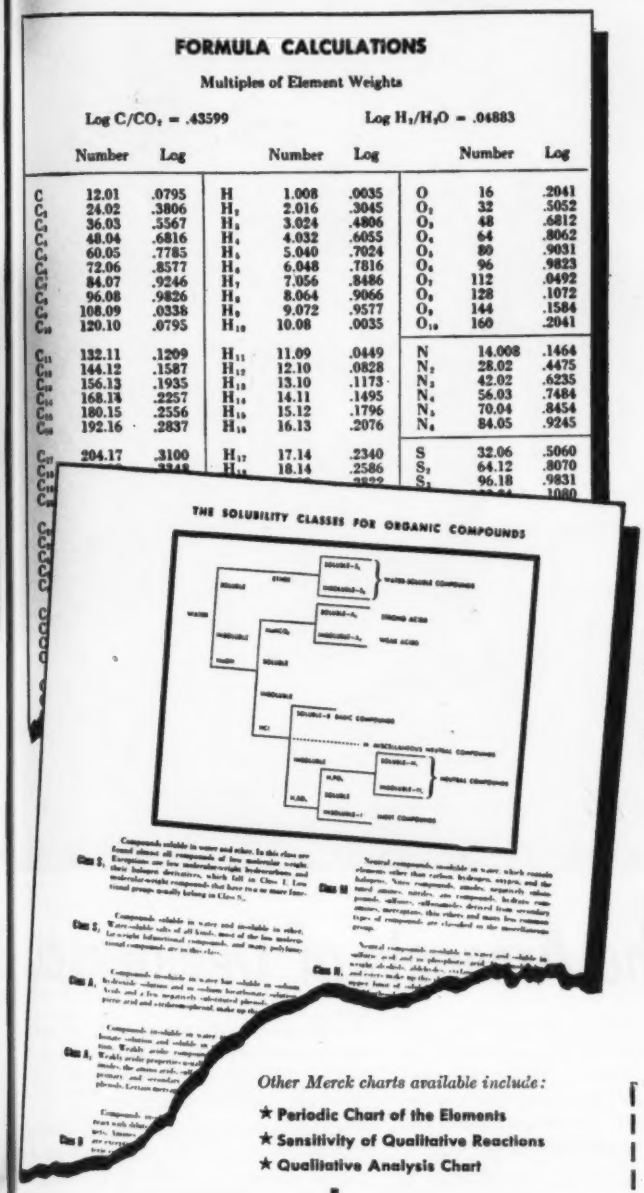

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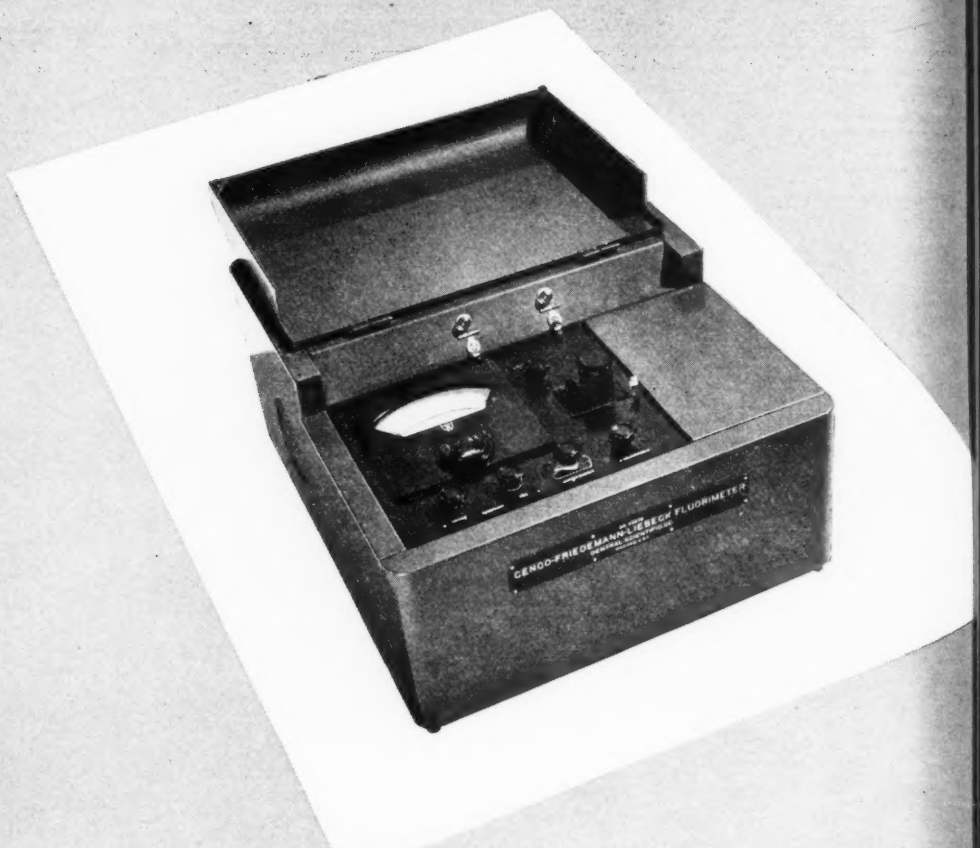

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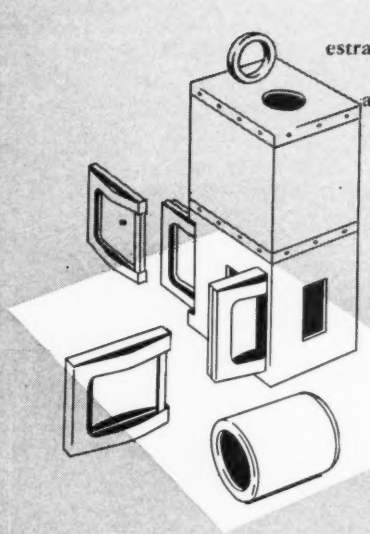

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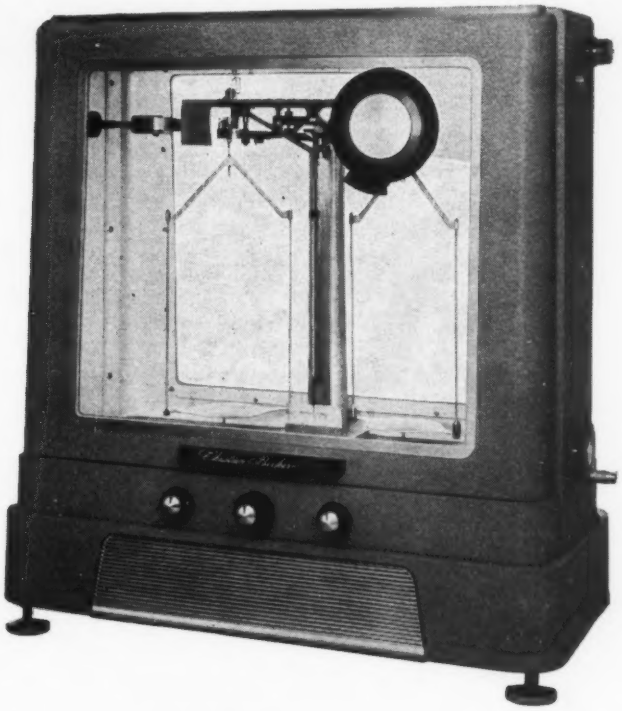

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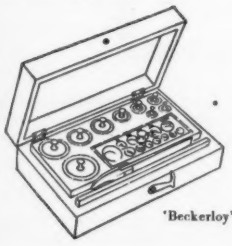

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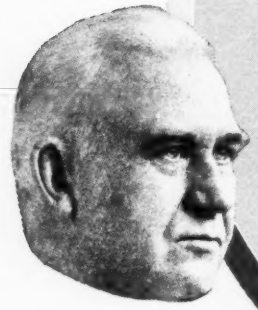

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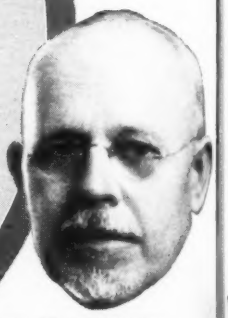

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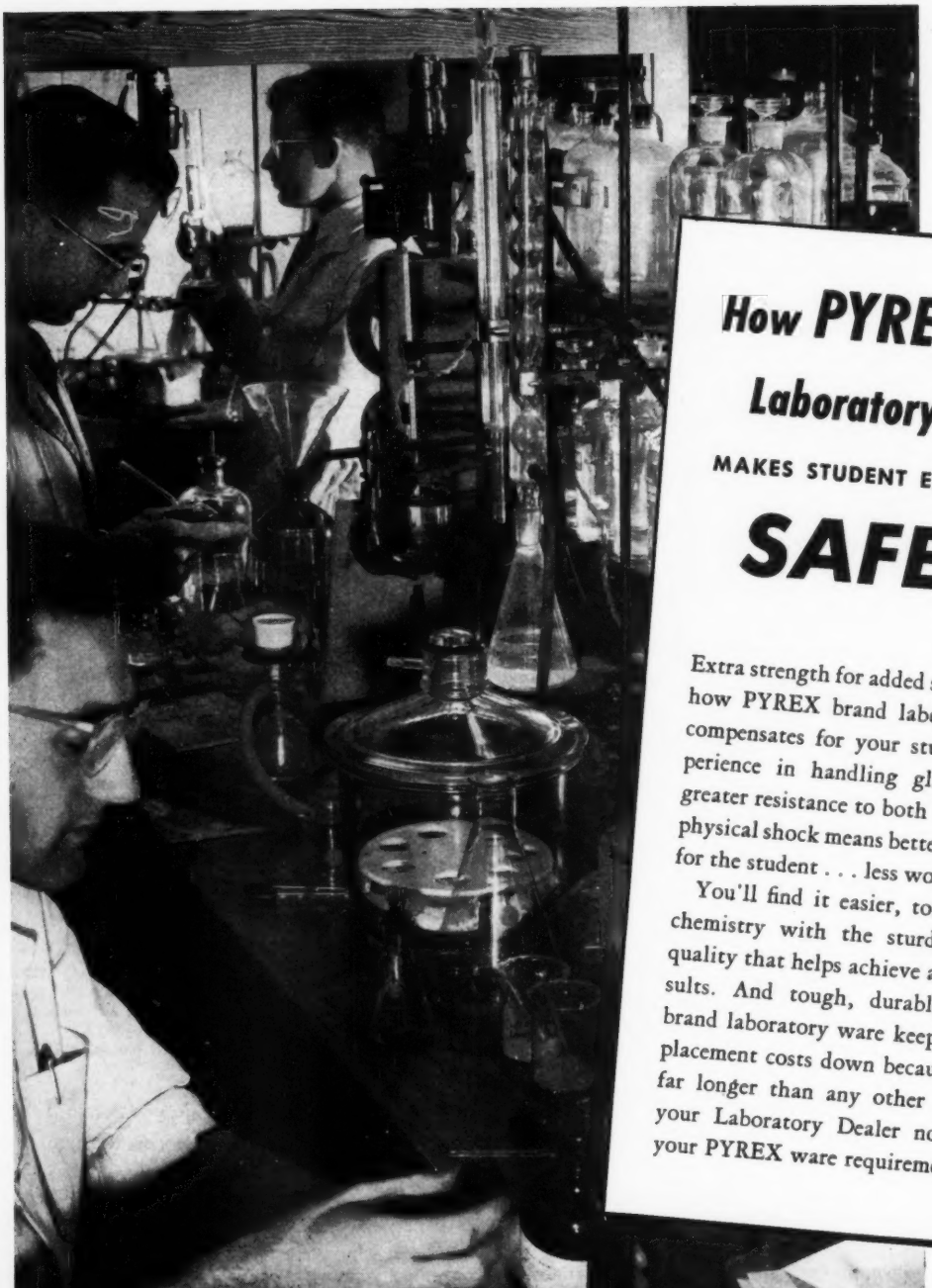

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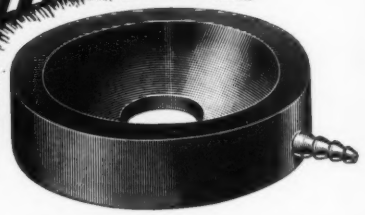

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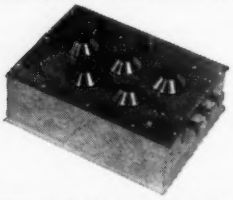

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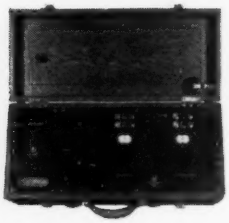

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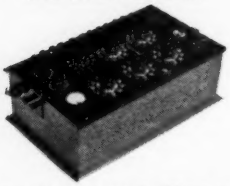

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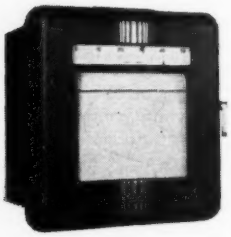

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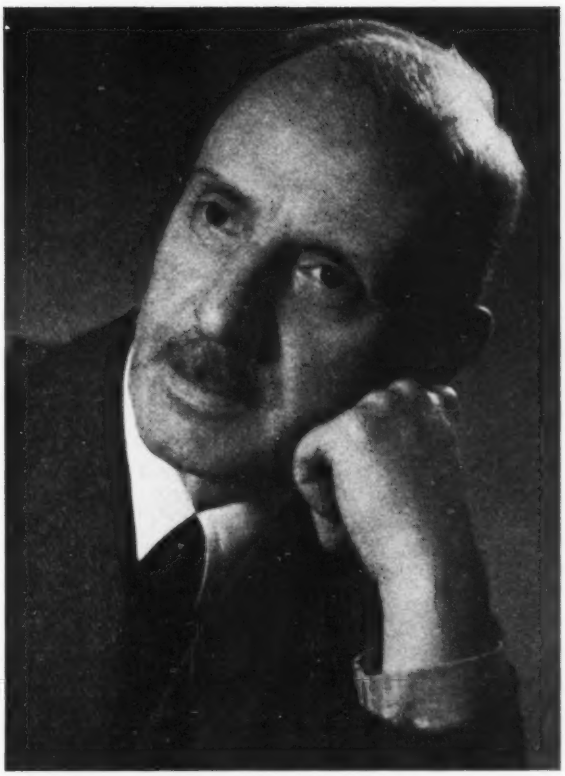

Rudolf Pummerer

(See page 243)

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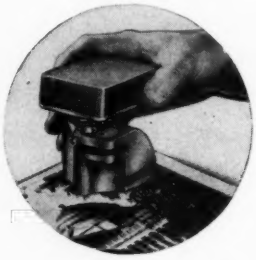

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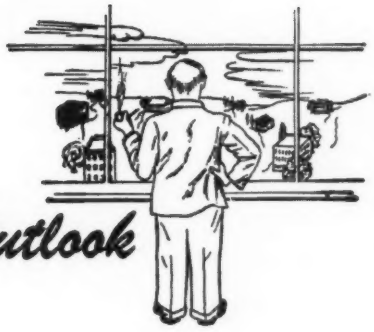


Editor's Outlook

In a recent article on "Teaching the Art of Research"[1] are some comments and suggestions on the planning of research which are well worth reading by young, aspiring scientists. After some discussion of the scientific method (to which I propose to revert at another time) the author launches into a discussion of the "strategy" and the "psychology" of research. First, why do people indulge in research?

The incentives in research are complex but the main ones are curiosity and the desire to win the approbation of one's fellows and the chief reward is the joy of discovery. The method by which the discovery is made is little more than a means to an end.

The author next refers to the many conflicting views on the desirability of controlled planning in research and the extent to which it should be carried.

Most of this confusion can be cleared up if one explains... that there are three different levels of planning. The first is the actual conduct of an investigation by the worker engaged in the problem. This corresponds with tactics in warfare. It seldom goes beyond the next experiment. The second level involves planning further ahead on broad lines and corresponds with strategy in warfare. Planning at this level may not be confined to the person engaged in the problem for often it also is the concern of the research director and the technical committee. Finally, there is planning of policy, the part played by the government in warfare. This type of planning is mostly done by the committee which decides what problems should be investigated and what projects or workers should receive support. Research can be planned but discovery can not.

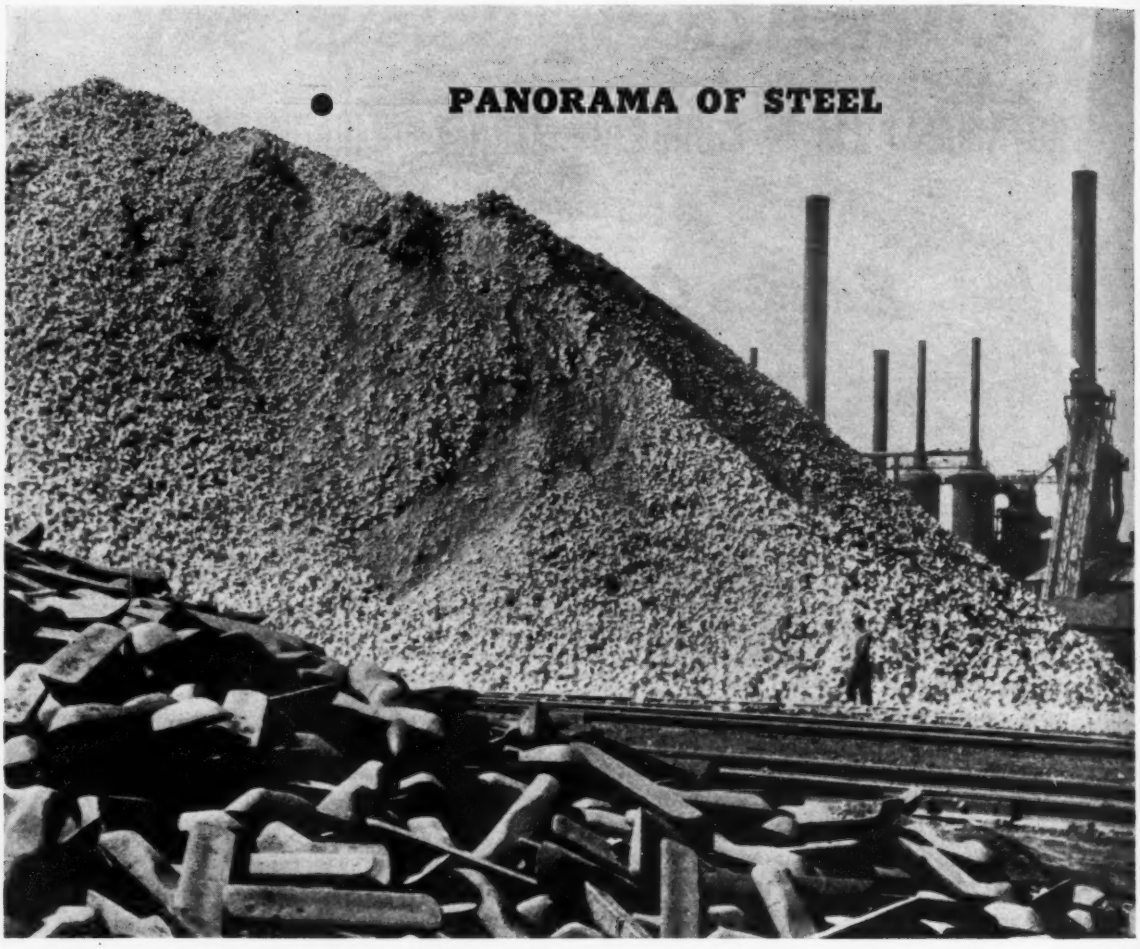
[1] Beveridge, W. I. B., *Research*, **4**, 97–101 (March, 1951).

The proper place of hypothesis is pointed out and the case of Charles Darwin cited, who said he could not remember a single one of his own hypotheses which did not have to be discarded or considerably modified. Along this same line it is suggested that:

The general strategy of research is to work with some clear objective but at the same time to keep alert for and seize upon unexpected opportunities. As soon as anything new is discovered one examines it from all points of view and relates it to previous knowledge. The discovery is the more intriguing if it does not fit readily with current beliefs, because further investigation may show that these are wrong and a whole new field may be opened up. The really exciting thing about discovery is not so much its intrinsic importance as its possible fruitfulness in leading to still more facts and theories. It is characteristic of successful investigators that they follow up each discovery until they exhaust all its implications.

A very close relationship is advocated between the planning and conduct of research and its continual assessment and evaluation.

A useful discipline that the young scientist should learn early is to write a report of his investigation before it is completed. If it is a long investigation, lasting over several years, a progress report ought to be written every six months or at least once a year. Writing a paper before the work is completed is a useful device for revealing weaknesses that can be rectified while the materials are still to hand; and it is much easier to write a paper well while one's memory of the work and literature is fresh and interest still active. However, early writing up does not necessarily mean early publication, for some restraint is often advisable before rushing into print.



Pig Iron in Foreground, Limestone in the Rear, Near Blast Furnaces

PANORAMA OF STEEL

CHARLES M. PARKER
American Iron and Steel Institute, New York, New York

THE iron and steel industry of the United States comprises 400 plants located in 250 communities in 30 states. Those establishments, manned by 620,000 employees, have capacity to produce more than 71 million net tons of pig iron and more than 100 million net tons of steel ingots. We have capacity to produce over 1300 pounds of steel for every person in the United States.

The industry is owned by some 640,000 stockholders whose total capital investment is over $5.7 billion or about $38 for every person in the United States; the capital investment per employee in the industry is approximately $6900.

The steel industry handles more materials per ton of finished product and in a greater variety of ways than any other large-scale manufacturing industry in the world. It has been estimated that for every ton of finished steel shipped, 68 tons of raw materials, steel-in-process, by-products, and waste products are handled. In addition, 150 tons of water are employed for every ton of finished steel produced.

To assist men of the industry in that unparalleled job of materials handling and production, the industry employs nearly 12 million mechanical horsepower or about 200 horsepower per worker.

The products of the steel industry are used by approximately 33,600 manufacturing establishments within the United States, a number nearly 65 per cent greater than was using steel in 1939.

The present rate of production is an annual tonnage of 100,800,000 tons. That means that every time your heart beats and pumps three ounces of blood, the steel industry produces 5500 pounds of steel.

Based on a paper presented at the Twelfth Summer Conference of the New England Association of Chemistry Teachers, University of Connecticut, Storrs, Connecticut, August 21, 1950.

EXTRACTION

Steelmaking in the United States starts on the iron ranges of Minnesota, New York, and Alabama; in the coal mines of West Virginia, Kentucky, and Pennsylvania; in the limestone quarries of Michigan and Pennsylvania; and in a myriad of other locations in every quarter of the globe.

Let us concentrate our attention for the moment on the Mesabi Range in Minnesota. From that range more than 1.6 billion tons of iron ore have been shipped to the steelmaking furnaces of America since the opening of the range in 1892. There is considerable difference of opinion as to just how long the reserves of Mesabi will last. I do not propose to offer an opinion one way or the other, but it is reasonably certain that before a great many years have elapsed, it will be necessary to extract from the submarginal ores of Minnesota the iron necessary to operate our furnaces, or to import it from abroad.

The amount of iron ore necessary to produce a ton of pig iron has risen in the past ten years. The top quality grades have been mined out and we are working with somewhat lower grades which contain less iron and more silica. Substantial tonnages of ore must be beneficiated by washing, screening, and sizing, and blending is a universal practice.

The primary effort of beneficiation is to reduce the silica content of ore to 9 per cent or less, and to increase the iron content to 60 per cent or more. For economic operation, recovery of iron by beneficiation should exceed 85 per cent.

The concentrating processes which are presently employed are reasonably simple. So-called wash ores are crushed and the excess silica is washed away by water. The problem of extracting iron from taconite, however, is more difficult.

Taconite is a hard rock which contains between 25 and 35 per cent of iron, mostly as fine grains of hematite and magnetite embedded in a matrix of silica. In some areas, magnetite prevails while in other areas, hematite prevails.

For millions of years nature has been leaching iron from taconites by the slow percolating action of water. Now, scientists of the steel industry propose to transmute the iron content of taconites into thousands of tons of valuable ore concentrates per hour by developing processes for grinding the rocky substance to powder and separating the iron particles from the mixture. What nature took millions of years to achieve, the mining industry now seeks to do by mechanical and chemical means in a single hour.

Unfortunately, nature has seen fit to play a little joke. Magnetite is magnetic and hematite is nonmagnetic. Magnetic separation is a rather simple, efficient operation. Separating the hematite is much more difficult, and the hematite content of some of the taconites is higher than the magnetite content.

In one operation where magnetite predominates in the taconite, the rock is ground and the magnetite is separated by magnetic means. This is an interesting operation in that reversal of polarity is used to rotate the magnetic particles and so shake off minute pieces of nonmagnetic gangue. The final concentrate which is 63 to 65 per cent iron and about 8 per cent silica is formed into balls or pellets which are heated to about 2000°F. in a continuous shaft-type furnace. The pellets are charged into blast furnaces in the same fashion as iron ore.

At the present time froth flotation methods are being developed to separate the hematite from types of taconite which must be ground to 65 mesh or finer. In one such method powdered taconite is introduced into soapy water through which air bubbles are blown. The iron particles attach themselves to the froth and rise to the surface while the sand sinks into the underlying water. Flotation methods are expensive, however, and economical methods are being sought to convert hematite to magnetite.

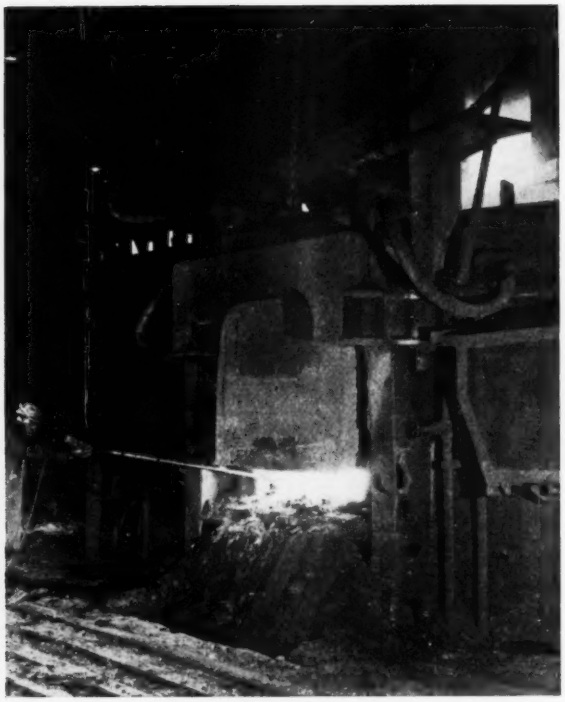
The physical condition of much of the iron ore now being taken from pits in the Great Lakes Region, and the physical condition of ores of all regions of the United States which must be beneficiated before use in blast furnaces, makes an agglomerating or sintering operation necessary. In addition, blast furnace flue dust which is high in iron content must be utilized efficiently and economically.

In sintering, finely ground ore is mixed in a thin layer with about 10 per cent of its weight of powdered coal or coke. The mixture is ignited and air is drawn through the mixture to promote combustion. The iron fuses and forms a strong cellular mass suitable for use in blast furnaces. This process makes possible the economic use of ore particles too fine for blast furnace use, and also permits the recovery and economical use of iron-bearing flue dust. The sulfur content of ore may be materially reduced in sintering.

SULFUR REMOVAL

I shall occasionally refer to the necessity of removing sulfur from raw materials and from molten pig iron or hot metal, as it is called in the mill, and to the efforts being made to prevent its entrance into steel. Sulfur exerts an adverse influence upon the plastic properties of hot steel, causes surface imperfections during manufacture which are costly to remove, and segregates in such a manner as to cause variations in mechanical properties from center to outside of an ingot. In turn, these variations exhibit themselves across the surface of a sheet or plate and cause difficulties or failures during forming, bending, and drawing.

Quality of coking coal has declined substantially in the past ten years. Reserves of high-grade coal have been depleted and mechanized mining methods have decreased the selectivity of mining and increased the quantity of slate and other debris brought to the surface. In some mines debris amounts to 25 per cent of material handled. Sulfur content has risen three-fold and ash content has doubled. Such conditions make necessary the use of increased quantities of limestone to flux off impurities, and that, in turn, makes necessary



Taking Temperature of Heat of Electric Furnace Steel with Immersion Pyrometer

higher coke consumption and decreased air volume blown into blast furnaces.

In order to counter those conditions the industry is washing and cleaning coal to rid it of debris and decrease sulfur. Washing removes about two-thirds of the inorganic sulfur present but removes none of the chemically combined organic sulfur. About 85 per cent of the sulfur in the steelmaking process enters the cycle in coke. Blending high and low volatile coals from many mines is also practiced to secure greater strength and greater uniformity and to permit the use of strip coal and anthracite fines.

In spite of efforts expended on coal and ore the sulfur content of much hot metal is too high for economic steelmaking. In the steelmaking process the most logical point at which to reduce the sulfur content of the metal is at the blast furnace stage, because most of the sulfur enters the cycle in the coke and ore.

At the blast furnace the steelmaker has the choice of attempting to remove sulfur from the hot metal as it leaves the blast furnace or attempting to remove increased quantities of sulfur in the blast furnace process by increasing the sulfur-carrying capacity of slag.

Methods have been devised to reduce the sulfur content of hot metal as it leaves the blast furnace, but in spite of the fact that such methods are chemically feasible they leave something to be desired, inasmuch as they cause objectionable fumes and require additional facilities and materials. Reduction of sulfur in the blast furnace would seem to be more desirable from both a metallurgical and operating point of view.

It is thought that under present methods of operation the sulfur-carrying capacity of slag may not be exhausted. Some work has been done but the mechanism and rate of transfer of sulfur from metal to slag is unknown. It is known, however, that experimental slags are very slow in reaching equilibrium, much slower than in reaching phosphorus equilibrium, and that fact may be highly significant in correctly devising experimental methods. Radioisotopes of phosphorus and sulfur are being used in this work.

Incidentally, high-speed color photography is being employed to study the physical reactions inside the hearth of a blast furnace. The pictures are taken at a speed of 3000 frames a second and show the travel of incandescent particles of raw materials which are estimated to be moving at a speed of 135 miles per hour. The pictures were made by sighting the camera through the tuyeres or air entry pipes of the furnace.

The problem of removing excess silicon from hot metal is analogous to the problem of removing sulfur. Excess silicon requires heavy limestone charges to prevent the acid-forming silicon from attacking the refractories of the basic open hearth furnaces. Moreover, large quantities of low silicon iron can be charged, thus improving furnace practice and yield.

A method of using mill scale, the iron oxide resulting from rolling mill operations, to reduce the silicon content of hot metal has been developed. The reaction is very rapid oxidation of silicon and reduction of iron, equivalent to the first minute of the Bessemer blow. The manganese reduced is recovered by using in the blast furnace the slag formed during the reaction.

FURNACE OPERATION

In the recent past there have been many statements in the public press concerning the air conditioning of blast furnaces and their operation at high top pressures.

In operating a blast furnace under high static top pressure, the amount of air or "wind" is increased approximately 18 per cent over normal operation, top pressure is increased substantially by throttling the exhaust gases, and blast pressure is increased by about 34 per cent. The chief proponent of the process claims that iron production is increased 11 to 20 per cent, coke consumption is decreased about 13 per cent, and flue dust is decreased 30 per cent. In spite of the proved validity of those claims, there are other blast furnace operators who believe that similar results can be achieved by less complicated and costly methods.

In operating a furnace with dry blast, moisture is removed from the blast by refrigeration, the theory being that the moisture tends to retard chemical reactions and wastes coke in decomposing the moisture.

Strong pressure by the government for more pig iron during the war forced a number of blast furnace operators to install dry blast apparatus before facts or figures could be developed by pilot installations.

Performance has been followed with great interest

and hope but up to now no significant measurable benefit has come from dry blast operation, particularly north of Birmingham, Alabama.

CHEMISTRY OF SLAGS

Metallurgy has sometimes been defined as chemistry at high temperatures. The chemistry involved concerns not only the metal itself but the materials by which the metal is brought to desired chemical composition and physical condition. The complementary materials form slag, the chemical behavior of which is of major interest in all phases of iron and steel manufacture. In order to improve steel quality or to develop new kinds of steel and new methods of steelmaking a clearer and deeper understanding of slags is necessary.

The chemistry of slags is dependent to an important extent upon their "basicity," a property which has not been clearly defined. One useful measure of basicity is "excess base" which is defined as the uncombined base, the sum of the oxides of calcium, manganese, and magnesium, after neutralization of silica, alumina, phosphorus pentoxide, and ferric oxide.

The concentration of the oxide ion in slag determines its basicity but up to now no direct means is available for measuring this concentration. Other ions present in slag are positive metallic ions such as calcium, and complex negative ions such as orthosilicates and metasilicates. An ionic theory of slags has been postulated. Equations have been set up to permit use of the theory in a quantitative manner for simple iron oxide slags and constants have been derived to satisfy equilibrium conditions. Further studies will endeavor to relate this theory to slags of a more complex nature.

USE OF OXYGEN

The use of oxygen in steelmaking has settled down to the point where it may be talked about as just another production tool. Oxygen is used in steelmaking in two entirely different ways: one use is to speed combustion of the fuel and the other is to oxidize the carbon in the bath.

Accelerated oxidation has two distinct advantages, the more rapid carbon drop saves time, the increased bath temperature increases the fluidity of the slag, undissolved lime goes into solution, and fuel is saved. In fact some plants report that they must reduce the fuel rate during accelerated oxidation. Moreover, less bank erosion is experienced in the open hearth furnace as banks are exposed to high oxide slags for a shorter time than in normal practice. The chief disadvantage of the use of oxygen in this operation is splashing of the slag which may erode the roof and front wall of the furnace.

The use of oxygen for decarburization is made use of most commonly by shops making low carbon steels. The reason for this is the normal slowing down of carbon elimination in the neighborhood of 0.12 per cent carbon. Oxygen speeds up the drop at these low carbons and maintains or increases the bath temperature. Its use is of particular value in this connection in the manufacture of low carbon stainless steels in the electric furnace.

In using oxygen to accelerate the combustion of fuel, oxygen diminishes the quantity of the products of combustion for the same weight of fuel burned, thereby increasing the flame temperature and decreasing the amount of heat carried away by the outgoing gases. This, therefore, permits the burning of a greater weight of fuel in a given furnace in a given time and the amount of heat transferred to the bath per unit of time is thereby increased.

The advantage in this use of oxygen is the decrease in heat time from charge to tap, but in order to take advantage of this rapid heat input without damage to the furnace the charge must, of course, be able to receive it. Cold scrap can absorb the heat as fast as it is made available. The general practice is, therefore, to use oxygen during the melt-down period or up to the addition of hot metal.

During 1949 at least one effort was made to use oxygen to enrich the air entering a blast furnace. The results of that experiment have not been published but it is known generally in the industry that the results given for similar Russian experiments were not duplicated. Recently a story came out of Europe that Russian engineers had tried to duplicate the experiments of Peter Kapitza and had failed. That may be at least one reason why Kapitza has been exiled or at least "sent to Coventry."

NEW PROCESSES

Experiments of great promise have been made with a slide-blown Bessemer converter known as the "turbo-hearth," which contains a magnesite lining. It is expected that by this method a low nitrogen, low phosphorus steel well suited for severe deep drawing operations may be produced.

The turbo-hearth combines the advantages of the basic open hearth and acid Bessemer processes, leading steelmaking methods used in the United States. The steel is made in a partly closed vessel lined with basic refractories. As in all pneumatic processes, heat is supplied by chemical action of blown air with carbon and other chemical elements in the charge, but in the turbo-hearth the air current is applied from the vessel's side onto the hot slag and iron of the liquid charge, rather than from the vessel's bottom through the entire charge. The action is fast and direct, but subject to controls which reduce the nitrogen and phosphorus contents, making a steel comparable with open hearth grades, suitable for more general use than conventional Bessemer steels.

For many years men have sought the art of continuously casting steel into a form suitable for direct rolling. Such a process, if successful, would eliminate ingot molds, soaking pits, and blooming mills. Moreover, it was hoped that elimination of the ingot would automatically eliminate the ills of the ingot: pipe, seams, blowholes, scabs, excessive columnar structure, burned or otherwise weakened corners, segregation of elements, and other such metallurgical abominations.

About six years ago two American steel companies joined forces to pursue the problem. In March, 1948, the first successful casts were made and 45 tons of product shipped to a commercial user.

The process consists in pouring regularly melted electric furnace steel into a transfer ladle which is lifted to the top of a 75-foot tower. There the steel is poured into an inductively heated holding and pouring ladle. The steel is poured into a long vertical mold which is water-cooled. Shortly after the skin freezes, the steel contracts and shrinks away from the mold. This permits downward vertical motion, thus permitting more liquid steel to enter the water-cooled area of the mold. It is obvious that a very fine coordination must be maintained between shrinkage rate and the lift which lowers the continuously forming mass of metal, called a billet for want of a better name.

The continuous casting process makes possible faster cooling of the molten steel, thus producing a fine, uniform crystalline structure with little segregation, few nonmetallic inclusions, and almost no serious surface imperfections. There still remain to be solved many problems in metallurgy and even more problems in mechanical engineering before this process can begin to compete tonnagewise with standard production methods.

In the rolling mills so many improvements have been made that, once again, it is only possible to touch on some of the more important ones. The electrification of the industry has proceeded very rapidly and the tendency in nearly all mills is to operate each unit, and indeed sometimes the many components of a mill, by individual electric motors. In this way higher speeds and more complete controls have been achieved.

The use of electronic controls and magnetic amplifiers is quite common. There are now in operation cold mills controlled by the regulating tensiometer, an instrument which automatically and almost instantaneously regulates the speed of one stand of rolls with respect to the next, and so makes possible better grain-size control and closer tolerances.

There are four high-reversing cold strip mills in which the back-up rolls are driven and the strip is rolled under high tension. These mills make possible the rolling of stainless and electric steels at approximately three times the rate of a conventional mill in which the steel is not subjected to deliberately added tension.

Strip steel is now being heated for rolling and annealed by induction which results in better quality. Continuous tin coating and galvanizing lines are in operation which produce a more adherent coat which will not flake or break in forming, and which is more uniform than the coating produced by conventional hot-dip methods. They also make possible the production of these quality materials in heavy oils for use on automatic fabricating machines.

Another new instrument in the tin plate field is a coating-thickness meter. It may be set for coatings of various weights, three hundred-thousandths of an inch being the usual thickness. If desired, the meter literally will sound an alarm the instant tin coating varies from uniform.

The X-ray thickness gage is used to determine the thickness of strip steel as it leaves the last stand of the finishing mill. Operation of the gage consists in setting the indicator at the thickness desired, setting the deviation index at zero, and inserting into the control beam a piece of strip of the desired thickness.

The gage operates on the theory that equal parts of an X-ray beam will be transmitted if beams from a common source of radiation are made to pass through pieces of like material and having the same thickness. Both reference beam and measuring beam emanate from the same tube. The reference beam continuously penetrates the control sample within the gage, while the measuring beam simultaneously penetrates a deviation control sample and the strip being rolled. The deviation sample is positioned automatically in such fashion as to maintain the relationship:

$$\text{Deviation} + \text{Sample} = \text{Produced Strip}$$

The accuracy and reliability of the X-ray gage are very good; moreover, it is not affected by changes in atmospheric conditions or other environmental factors and it requires a minimum of checking and adjustment. A similar instrument employing gamma rays is in use.

The practical development in the United States of cold extrusion of steel can be traced to a German process. The Germans extruded only thoroughly deoxidized low carbon steel but the process is being used in this country for higher carbon steels as well as alloy steels containing chromium, nickel, manganese, and molybdenum.

In theory, most steels are quite plastic, even at ordinary temperatures, and therefore can be extruded cold. An important factor in the cold extrusion process is the use of a crystalline metal phosphate coating which forms a lubricating film so strong that it remains unbroken under tremendous pressure.

Generally, cold-extruded shapes have a fine finish, hold close tolerances, and exhibit high mechanical properties. Increases in strength resulting from extrusion have made possible the use of thinner sections for many applications, and as a rule finished parts represent a 90 per cent yield of original stock weight.

On the other hand, cold extrusion requires about twice as much power as drawing, and the stresses which can be withstood by the extrusion dies must be taken into account as an important limiting factor. Nevertheless, solid and hollow tubular shapes produced by cold extrusion are being used, in several instances, to replace forgings and castings and to reduce waste in screw machine parts.

TESTING TECHNIQUES

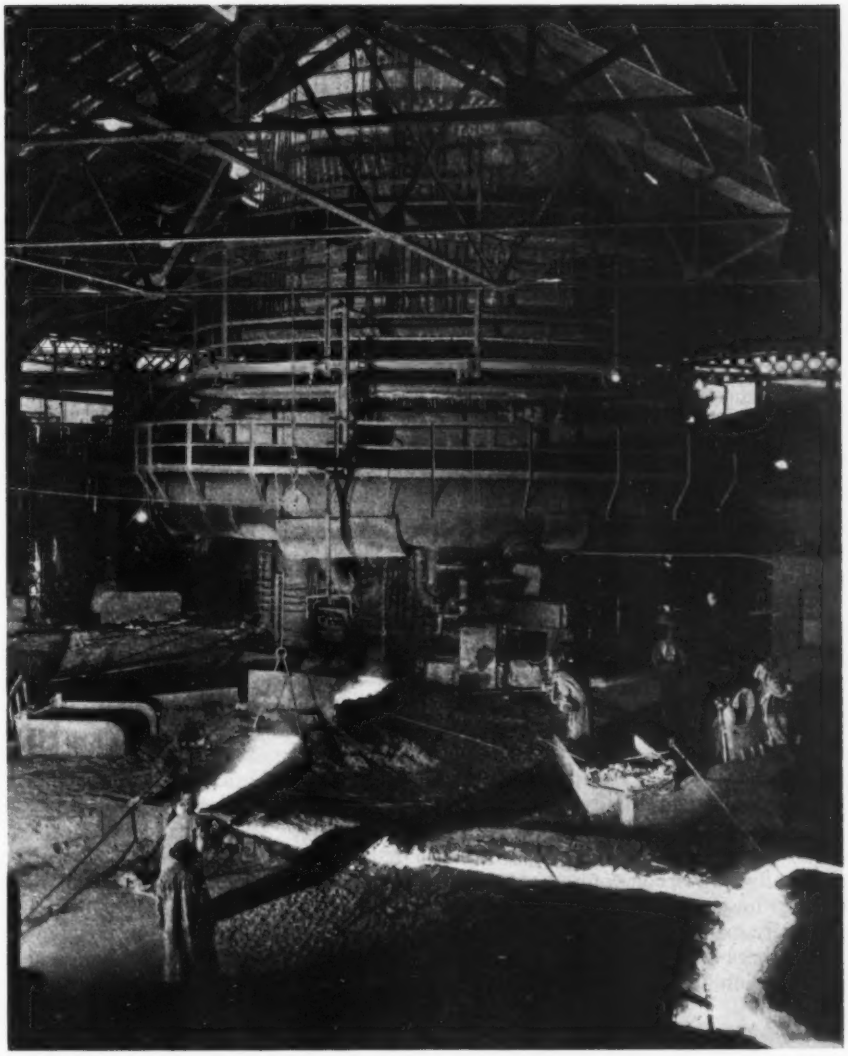
Sound made visible is one of the newest tools used by metallurgists in their never-ending efforts to improve steel quality. Because supersonic waves are not audible to the human ear, the waves are made visible by an arrangement of electronic tubes.

It is well known that the intensity of sound varies with the material through which the sound waves pass. If a supersonic wave passes through a piece of steel which is free of all defects a steady wave will be reflected. If, however, there are cracks or holes in the steel, or if it contains nonmetallic inclusions which differ in density from the steel, the reflected sound waves will be distorted and the defect detected.

Knowing the length of the supersonic waves and number per second, it is possible to calibrate the instrument and locate the defect in the steel within very close limits.

Another new testing technique makes possible the determination of the hardness of single crystals of steel. Known as the "microhardness tester," the instrument is capable of taking hardness readings at three spots within the width of a human hair.

Like other hardness testers, but on a much finer scale, this machine marks a polished sample through the action of a diamond four-sided pyramid under a carefully controlled load. The depth of the mark, measured in microns (0.000039 in.) is an index of the hardness of the steel. This test is more important in studying segregation of elements, the effect of shape and section size on mechanical properties, and the effects of heat treatment.



Cast House Blast Furnace Showing Iron in Runners Going to Ladles from the Furnace

Sometimes very simple, homely things are responsible for major advances in science and technology. Take for example the lithographed top of the humble catsup bottle. It appears to be a simple thing but it is not. The method of fabrication used to manufacture it requires steel of uncommonly good homogeneity and the ability to be stretched equally in all directions. Until a few months ago there was no test known which would predict performance before actual trial of material.

A new instrument was devised which is known as a "recording torque magnetometer." That instrument contains two electromagnetic coils into the fields of which a steel sample about an inch in diameter is introduced. The sample rotates in the field and finally comes to rest in a position in which the grains of steel are most easily magnetized. The angle through which the sample rotates is a measure of "preferred orientation" and therefore a measure of the ability of the steel to deform uniformly under the action of drawing dies. Least angle of torque is most desirable. If the steel were completely homogeneous and the crystals all equiaxed, the sample would not turn at all, and the steel would be ideal.

NEW STEELS

Even "show business" comes to the steel industry for assistance. Hillbilly singers who wanted to reach an octave above middle "C" on the tenor banjo and tenor guitar desired a string which would permit such an

extension of their range. An "A" string was developed which has a tensile strength of 460,000 pounds per square inch, more than twice the strength of the wires which comprise the cables which support the main span of the San Francisco Bay Bridge.

The banjo wire, which is plain high carbon steel, is produced by a special combination of heat treatment and especially long and exacting cold working procedures. Only 0.010 inch in diameter, each wire will take a stress of nearly 38 pounds.

New types of free machining steels have been developed which increase production up to 300 per cent over ordinary types of high sulfur free-cutting steels. Employing only the elements carbon, manganese, phosphorus, sulfur, and silicon, the new steels are processed in such a fashion as to render the sulfide inclusions globular rather than elongated or fibrous as was previously thought necessary to produce free-cutting characteristics.

It is interesting to know that this development was approached by two different companies from two completely different directions to produce the same end result—globular sulfides—and that patent applications were filed less than 24 hours apart.

An anticipated shortage of the comparatively rare alloying element, columbium, or niobium as it is now officially known, led to the development of a new low-carbon stainless steel of wide industrial use. Carbon in stainless steel is an element of vulnerability when the steel is subjected to conditions which promote intergranular corrosion. Up to a few months ago niobium or titanium were employed to minimize that vulnerability, particularly at elevated temperatures.

Now, methods have been perfected to reduce economically the carbon content of such steels to 0.03 per cent or lower. Since that amount of carbon exists in interstitial solid solution in the steel, intergranular corrosion, because of carbon, does not occur and the necessity for niobium and titanium vanishes.

MANGANESE

Next to iron, manganese is the most important element which is found in all grades of steel. Unfortunately its nature is such that there is no important quantity returnable from scrap to assist in relieving shortages, and even the most effective conservation measures can do little to extend the life of stocks on hand because proper and adequate use is essential to maintain steel quality. The iron and steel industry uses over 90 per cent of all the manganese consumed in the United States and more than 90 per cent of this country's total requirements are normally imported from distant sources.

A survey of the manganese contained in the slag from the industry's basic open hearth furnaces has indicated that the amount of manganese in these slags is roughly equal to the manganese used by the steel industry in the form of ferroalloys.

The major technical problem in the recovery of manganese from these slags is the separation of manganese and phosphorus. Up to now, no one has succeeded in separating those elements by any process capable of dealing physically and economically with 4 to 5 million tons of raw material. Many research programs are in progress in an effort to solve the problem, among them, one sponsored by the steel industry at the Bureau of Mines Experiment Station in Pittsburgh. It is still too early to report more than satisfactory progress.

CONCLUSION

The foregoing has been a brief and possibly oversimplified report of some of the more important problems which have been dealt with by the steel industry within the past two or three years—there are countless others under consideration, and countless more still to be dealt with. There is no doubt in my mind concerning their solution—our people will see the job through to success as they have done so many times in the past. They are the most competent steelmakers in the world.

WORKSHOP

The science faculty of Mundelein College, Chicago, will sponsor a Secondary School Science Teachers' Workshop, June 18–22. Two lectures each morning will be offered by the College faculty in each of three fields: biology, chemistry, and physics. The afternoon will be devoted to seminars conducted by secondary school teachers of science. The program of lectures to be offered was determined by a questionnaire sent to all Chicago area Catholic school science teachers as well as those in the Congregation of the Sisters of Charity of the Blessed Virgin Mary teaching in all parts of the United States, for whom the workshop was designedly planned. Sister Mary Martinette, B.V.M., is chairman.

RUDOLF PUMMERER

RALPH E. OESPER
University of Cincinnati, Cincinnati, Ohio

RUDOLPH PUMMERER perfectly exemplifies the university professor who has devoted his entire career to teaching chemistry, training chemists, and turning out first-class research. Imbued, as always, with the importance and dignity of his chosen life work, the approaching close of Professor Pummerer's academic life led him to write recently: "The manifold collaboration and other contacts with the student body constitute one of the chief joys of the teaching profession. Sharing their troubles, smoothing the way to creative scientific activity or to practical work in industry, seeing their eyes light up at the first discovery, these are the things which intimately bind teacher and student together into a life-long fellowship, whose cultivation was always close to my heart. With sadness I look ahead to the day when the age limit will put an end to this activity and sever this association with young people, which most effectively keeps the teacher young in spirit."

Rudolf Pummerer was born on June 26, 1882, at Wels in Upper Austria. The family was then in very comfortable circumstances; the father had a profitable business producing illuminating oils by pressing rape and linseed. The boy's interest was early turned to natural history in the large garden attached to the home. In the Gymnasium at Kremsmünster he received excellent instruction in mathematics and science; the chemistry course, which included experiments, made a lasting impression of the lad of thirteen. He played the piano quite well, and was good at sports.

In 1896 his father was obliged to sell his factory because of the irresistible competition of electric lighting, and the family's finances suffered greatly. They moved to Munich where the chief emphasis in the Gymnasium was on the Greek, Roman, and German classics, but no chemistry was taught. Concerts, plays, and travel broadened the boy's outlook and increased his language abilities (English, French, Italian). He also became a tennis star, winning over one hundred prizes in Munich, Vienna, Prague, and from 1900 to 1905 he was in a class by himself in Bavaria.

In 1900 he enrolled at the University of Munich; his objective was chemistry. This resolve was definitely strengthened after he had listened to Baeyer's lectures for two weeks. In contrast, Röntgen's lectures in general physics struck no responsive chord. The nature and behavior of matter have always interested him more than nonchemical forces and mechanically motorized apparatus. Besides Baeyer, those responsible for his training were K. A. Hofmann (inorganic, analytical), R. Willstätter (special organic), and H. Wieland (organic) who was then giving his first lectures on terpenes. The doctorate thesis, dealing with pyrone, was prepared under Willstätter's guidance; the degree was conferred *summa cum laude* in 1905.

Despite his very limited financial resources Dr. Pummerer wished to devote himself to research. Even though he had no assistantship he began to work by himself (Willstätter had just received a call to Zurich). One day, Baeyer called him in and told him that a position was open at the Badische Anilin und Soda Fabrik at Ludwigshafen. "What do you wish to do?" "I want to do research." "What will you live on?" "I'll try to make some money on the side, perhaps by writing abstracts for the *Zentralblatt*." "You cannot live on that. I advise you to go to Ludwigshafen. If it does not suit you there I will always welcome you back here."

Dr. Pummerer was in industry for two and one-half years and worked in the indigo laboratory under Knietsch, Seidel, and Holt. Friedlaender, in Vienna, had discovered thioindigo and had assigned it to Kalle and Company for development. The B.A.S.F. also wished to enter this field. Pummerer discovered thionaphthaquinone, its oxime, and later its α-anil, all of which served in the production of sulfur-bearing indigoids. Twelve patent applications resulted. Although his prospects were bright, he resigned and returned to Munich. He supported himself by industrial research and carried on his main scientific work in his free time. By 1911 he had accumulated enough results to habilitate as Privatdozent, with a dissertation on "Studies in the indigo group," which, however, did not include his Ludwigshafen investigations.

In 1914 he received the appointment previously held by Wieland in chemical technology. When the war started he was put in the infantry, but from 1915 on he was in the chemical service. In the powder factory at Ingolstadt he learned much of value for his courses in technical chemistry. In 1917–18 he worked in Haber's laboratory at Berlin-Dahlem, where he studied the use of synthetic rubber in order to improve gas masks.

In 1919 he returned to Munich where the university laboratory was now under the headship of his revered teacher, Willstätter. The latter offered him a position, its chief duty being to assemble an elaborate exhibition collection of chemical technological specimens, etc. This provided a modest financial support for his growing family and enabled him to continue his researches.

In 1921 he was appointed head of the organic division of the Munich laboratory and his long-desired ambition was thus fulfilled. However, the prospect of entirely independent action induced him in 1923 to accept a call to the University of Greifswald as head of the chemistry

department. Two years later (1925) he again transferred, this time to the University of Erlangen, where he succeeded Otto Fischer as *ordinarius*, a post which he still occupies with his characteristic competency.

Professor Pummerer's researches have been almost entirely in the organic field. They have dealt with a variety of main topics, and their results have been important to both chemical theory and industrial practice. He has always been primarily interested in structural and valence problems and the dissection of reaction mechanisms, and he has become well known for his ability to get to the heart of intricate reactions, such as the Sandmeyer indigo process, the formation of levulinic acid from glucose, the dehydrogenation of phenols. More than one hundred important papers have come from his work, but he insists that the majority of these contributions were made possible solely through the able collaboration of his students and assistants. Physical weaknesses since 1918 have prevented him from personal preparative labors, and he has developed very intensively the Munich tradition of making test-tube trials at the desk of his collaborators. By this means he has kept in close touch with the real progress of the research. His lectures have always carried a strong reflection of his technical experiences, because he feels that both future teachers and industrial chemists should have a clear picture of the practical side of chemistry.

Professor Pummerer has been editor of the well-known Ahrens' "Sammlung Chemischer und Chemisch-Technischer Vorträge" since 1934 and of the *Zeitschrift für Angewandte Chemie* since 1947.

ANALYTICAL CHEMISTRY AND THE COOPERATIVE PLAN

JAMES F. CORWIN and A. R. WILSON
Antioch College, Yellow Springs, Ohio

THE cooperative work-study program has long been recognized as a major factor in engineering and business administration education. The University of Cincinnati has had such a program operating since 1911. The University of Detroit has been running their Engineering College cooperative program since 1917. There has been little evidence of a concrete nature, however, to show how well the cooperative work program supplements and implements a course in analytical chemistry. At Antioch College, where this type of cooperative program has been used since 1921 in a liberal arts institution, we have found that this program both supplements and extends the training in analytical chemistry usually required in college.

Briefly, the pattern of this program is as follows. The chemistry course is five years in length. Usually, there is one year of full-time study (40 weeks); and four years of cooperative study (20 weeks of academic work, 26 weeks of actual work). This means that the student, at the end of his course, has two full years of experience, or approximately 4000 hours of industrial laboratory experience. Since it is the policy to place the student on a job which is, in so far as possible, commensurate with his academic advancement, this means that, in many cases, he works in four different laboratory situations during his college career. In actual practice this is not always the case, since employers often wish to retain the more advanced students in hopes of making them permanent employees.

Since the student is not committed to stay with one employer for a period of more than six months the program attains a flexibility which works always to the advantage of the student. This is not to say that the student may not remain with one employer for a longer period of time, or even for his entire college course. It does, however, give him a bargaining power which allows him to obtain more advanced experience in line with his advanced academic standing, or to move to a position with another employer where this experience may be gained.

In other words, as the student advances in his knowledge of chemistry he is able to advance also in the

TABLE 1

Years in school	*Number of students*
3	4
4	17
5	9
6	4
Total	34

Average number of years per student 4.3.

TABLE 2

Field of interest	*Number of students*
Metallurgy	3
Organic chemistry	8
Tech. librarian	1
Biochemistry	4
Analytical chemistry	3
Physical chemistry	8
Chemistry teaching	1
Chemical engineering	2
Chemistry and business	3
Inorganic chemistry	1

TABLE 3

Type of analysis	*Description*	*Number*	*Number per person*
Volumetric	Noninstrumental	296	8.7
Gravimetric	Does not include moisture and ash	80	2.4
Colorimetric	All types	37	1.1
Instrumental	All types	108	3.1
Miscellaneous	All types used for analysis	264	7.9
Total	All types	785	23.2
Min. reported by one person			12.0
Max. reported by one person			60.0

responsibility and technical content of the job which he is performing under the cooperative work program.

The records of 34 students were examined for the effect of this program on their experience in analytical chemistry. The survey was made when these students were registered for Quantitative Analysis II, since this course is designed to complete or supplement each student's experience in analytical chemistry. The course is usually taken during the fourth or fifth years, but some returned soldiers are allowed to substitute army experience for cooperative work and, because of this, registered for the course during the third year. On the other hand, some students, for financial reasons, had elected to spend six cooperative years in school rather than four with one full-time year. Table 1 shows the distribution between the number of students and years in school. The total number of jobs held by these students during their tenure prior to the survey was 96 with an average of 2.8 jobs per person. Table 2 is a classification of the students as to interest. The types of analytical experience are classified in Table 3 under five general classifications common to analytical chemistry. These do not include repetitions of the same methods, sampling procedures, or standardizations. A breakdown of the three largest groups into subclassifications indicates the scope of the experiences and shows the type of methods commonly found in industry. These distributions are contained in Tables 4, 5, and 6.

These summaries represent students interested in all phases of chemistry and their industrial positions are often those that do not include much analytical work. For those interested in analytical chemistry, Table 7 represents a typical series of job experiences.

This survey shows that in general the cooperative work program lends itself very well to training students in analytical chemistry and allows them to come in contact with many methods and sampling procedures which are not available in the usual college course in analytical chemistry. It allows them to use instruments with which, normally, they would not come in contact until graduate school. The program lends itself especially well to those students interested in analytical chemistry as a career.

TABLE 4
Distribution of Volumetric Experience

Type of analysis	*Number*	*Number per person*
Acid-base	149	4.4
Direct	106	3.1
Indirect	43	1.3
Oxidation and reduction		
Permanganate	110	3.2
Dichromate	15	0.4
Iodimetric	59	1.7
Volumetric precipitation	37	1.1

TABLE 5
Distribution of Instrumental Analysis

Type of instrument	*Number of students using instrument*
Conductivity methods	4
Titrimeters	16
Polarograph	10
Spectrophotometer	14
Spectrograph	2
pH meter	34
Refractometer	14
Polarimeter	7
Flame photometer	3
Microscope-visual	3
Microscope-electron	1

TABLE 6
Types of Miscellaneous Analyses Which Two or More Students Have Used

Sieve analysis	Softening point
Specific gravity	Turbidity
Solubility	Boiling point
Melting point	Iodine number
Viscosity	Distillation range
Fractional distillation	Water analysis
Membrane permeability	Pyrometry
Extraction	Van Slyke—gas
Karl Fischer—moisture	Dumas nitrogen
Flash point	Chromatography
Fire point	Kline and Acree method for dextrose and levulose
Gas analysis	

TABLE 7

Year in school	*Courses taken during year*	*Type of company*	*Type of analytical work*
1	Inorg. chem., qual. anal.	Full year in school	No job
2	Quant. anal. I, org. chem. I	Paint Mfg. Co.	Control work, physical testing
3	Org. chem. II	Pharmaceutical Co.	Analytical organic
4	Phys. chem. I, quant. anal.	Chemical Mfg. Co.	Control work on 90 different products
5	Phys. chem. II, adv. anal. chem.	Same Chemical Mfg Co.	Developing analytical procedures

PILOT PLANT STUDY OF FLUORINE AND ITS DERIVATIVES

F. R. LOWDERMILK, R. G. DANEHOWER, and H. C. MILLER
Pennsylvania Salt Manufacturing Company, Wyndmoor, Pennsylvania

ELEMENTAL fluorine was discovered in 1886, and an impressive volume of experimental work on the preparation and characterization of fluorine compounds was done in the years that followed, but it was not until about 1920 that a realization of the commercial potentialities of this element initiated a steadily intensifying interest in the production and utilization of fluorine which was to have far-reaching results during World War II. The vital part that fluorine played in the separation of uranium isotopes by the diffusion process (*4*) and in the production of chemically inert lubricants and heat exchange mediums for the Manhattan project (*1, 14*) has been revealed.

Fluorine, a very pale yellow gas that condenses to a yellow liquid at −188°C., is the most reactive chemical element. It combines directly or indirectly with all of the other elements except the inert gases. Because of this great chemical activity, fluorine has been a difficult and dangerous material to handle. The rapid technological developments of the past eight or ten years, however, have eliminated many of these hazards and permit the safe handling of fluorine if a well-defined code of safety precautions is observed.

The method of producing fluorine today is fundamentally the same as that used by Moissan, who first prepared the element by electrolyzing anhydrous hydrogen fluoride containing dissolved potassium bifluoride. However, numerous technical difficulties had to be overcome before the operation of pilot plant or commercial scale cells could be made continuous and practical (*2*). Initial accomplishments in the development of fluorine cells by the research organizations of a number of chemical manufacturers were described in the Fluorine Chemistry Symposium held in September, 1946, and published in the March, 1947, issue of *Industrial and Engineering Chemistry*. Among these was the Pennsylvania Salt Manufacturing Company's diaphragmless laboratory cell (*6*) from which the pilot plant unit, described here, was developed. Actually, the pilot plant cell on which our engineering studies were made was of commercial capacity and has since been used in production operations.

The greatest activity in the chemistry of fluorine and its immediate derivatives must be considered as still in the research and developmental stages. Research continues, for example, on the preparation and use of fluorocarbons and on the use of fluorine and halogen fluorides in combustion systems, such as the hydrogen-fluorine torch (*11*). As exceptions to this generalization, the wartime application of the fluorocarbons (*1*) and the manufacture and use of sulfur hexafluoride (*13*) have attained an industrial status.

FLUORINE

Pure anhydrous hydrogen fluoride (a colorless, corrosive liquid which boils at 19.5°C.) does not conduct and is therefore not decomposed by an electric current. However, many substances, when dissolved in hydrogen fluoride, render that liquid electrically conducting. Potassium fluoride and bifluoride have been the most satisfactory readily available chemicals for this purpose. In electrolysis, the hydrogen fluoride is decomposed with the liberation of fluorine at the anode and hydrogen at the cathode. Hence, during operation, make-up HF must be supplied continuously or at intervals.

Fluorine cells are generally of three types. The low-temperature cell, of which the Moissan cell is the prototype, operates at temperatures below 50°C. and requires a high ratio of hydrogen fluoride to potassium fluoride in the electrolyte. The high-temperature cell, which was used in Germany during World War II, used an electrolyte approximating KF.HF in composition and operated at a temperature of about 250°C. United States practice has almost exclusively favored an intermediate operating temperature (80–120°C.) for which an electrolyte having a composition close to KF.2HF is most suitable.

Fluorine Cell. The body of the cell developed by Pennsalt is made of low-carbon hot-rolled steel plate welded into a rectangular tank 104 in. long, 16 in. wide, and 33 in. high (internal dimensions). The cells were stress relieved, welds were tested for slag inclusion by the Magnaflux method, and the interior surface was cleaned and freed of scale by acid pickling. The cell is equipped with a hot-water jacket to permit raising the electrolyte to operating temperature (100–105°C.). When the cell is operating, the heat developed requires that cooling be applied; this is accomplished by the circulation of thermostatically controlled water through the jacket. The cell cover, also of mild steel plate, is insulated from the cell body and "floats" electrically at the neutral zone of the cell. This cover is slotted to receive the anode assembly which is a battery of eight pairs of 2 × 5 × 24 in. or four pairs of 2 × 10 × 24 in. ungraphitized electrode-carbon blocks disposed along the principal axis of the cell. The anode shoes are

shrunk onto the carbon blocks to provide the best electrical contact and to minimize strain on the carbon. Two cathodes, paralleling and on opposite sides of the anode assembly, were constructed by welding horizontal iron strips at an angle onto vertical straps to provide a louvered cathode which directs the liberated hydrogen upward and away from the anode. High and low electrolyte level control and warning devices and gas pressure regulating and warning devices are provided. The gas space above the electrolyte is divided by a steel barrier which is attached to the cover and which extends about 3 in. below the surface of the electrolyte. This barrier effectively separates the fluorine, which flows up the anode into the central gas space, from the hydrogen, which collects in the outer space. An outstanding feature of this type of cell construction is the fact that a diaphragm is unnecessary and hence a part which is subject to corrosion and periodic replacement is eliminated. To the cell cover are attached nickel thermocouple wells for temperature control of the electrolyte, and self-reseating explosion ports. The latter features were incorporated during the exploratory phase of the work and were retained as an added safeguard even though many months of smooth operation indicated that they are unnecessary. Fluorine leaves the cell through a steel or copper manifold equipped with a current break.

Two of the cells described are shown in Figure 1. At the left is a spare cell cover showing the bus bar and its supports and the anode connectors. The view of the cells shows explosion ducts running the length of the cell and, at the far end, the connecting ducts which lead from the explosion ducts to the disposal tower. The cone-topped cylindrical chamber at the rear is the fluorine disposal unit in which waste gases are burned with city gas. At the near end of the cells are seen the two hydrogen outlets (light-gray inverted U's) with their rubber-hose current breaks. Directly in front of the rubber sections is the hydrogen fluoride make-up line with its valved branches to either side of the cell. Still lower is the fluorine exit line with its flanged connection to the cell. The current break in the fluorine line was effected by means of a special nonconducting inorganic cement. The short vertical section of 4-in. pipe in the fluorine outlet line is a trap to remove electrolyte spray.

Each cell has a current capacity of 2000 amperes, an anode current density of 70 amperes per sq. ft., and operates at 10–12 volts. The current efficiency has averaged 95 per cent. The output of each cell operating at 2000 amperes is 2.95 lb. of fluorine per hour. In a pilot plant demonstration of continuous operation (except for infrequent brief shut-downs for repairs, for anode replacement, or because of anode polarization), 4,700,000 ampere-hours of fluorine generation (6000 lb.) was obtained with two cells. Fluorine was bottled in steel cylinders or used directly for the production of chlorine trifluoride, sulfur hexafluoride, and metallic polyfluorides.

The fluorine leaving the cell will contain 4–5 per cent of hydrogen fluoride due to its volatilization from the electrolyte. After this is removed in the purification system, the fluorine gas will normally contain up to 1.5 per cent of oxygen and 1.3 per cent of inert gases.

Purification System. In the pilot plant, both the fluorine and hydrogen purification systems, which remove and recover hydrogen fluoride from these gas streams, consist of two 6-ft. towers made of 6-in. IPS pipe, reduced to 1 in. on both ends by steel flanges to accommodate the inlet and outlet piping. A screen holder and screen are provided to support the charge of $^3/_8$-in. sodium fluoride pellets. When on stream, sodium fluoride absorbs and reacts with the hydrogen fluoride, forming sodium bifluoride.

$$NaF + HF \rightleftharpoons NaHF_2$$

When the tower is spent, it is purged with nitrogen and

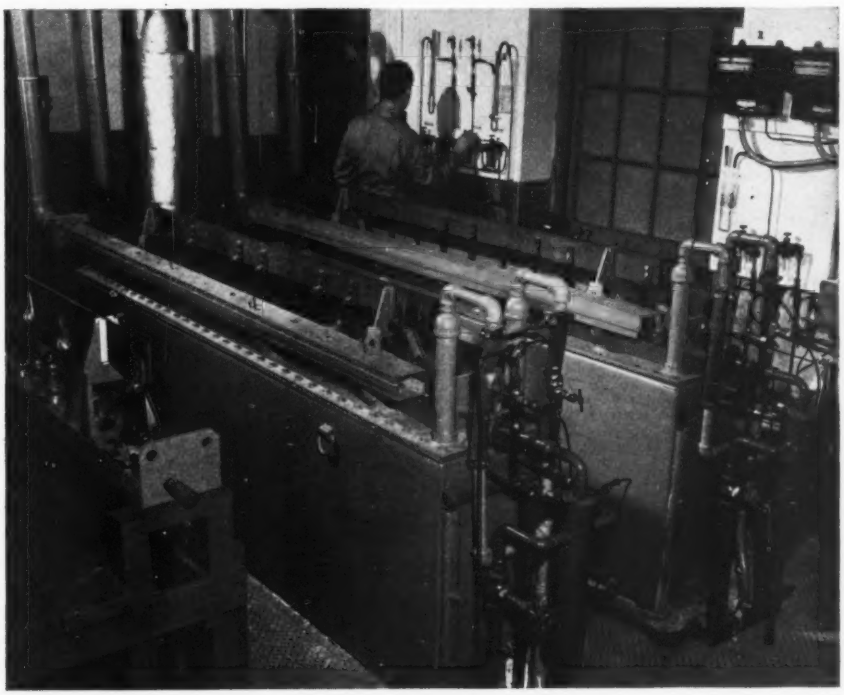


Figure 1. Pennsalt's 2000-Ampere Fluorine Cells

the fluorine-containing waste gases are piped to the fluorine disposal burner. The charge is then regenerated by heating at 300°C., at which temperature the bifluoride is reconverted into sodium fluoride for re-use in the next cycle, and hydrogen fluoride which is condensed and stored for re-use as cell feed. The heat required for regeneration is supplied by electrical strip heaters strapped to the outer surface of the tower and thoroughly insulated. Other forms of heating, of course, would be equally effective.

In Figure 2, the first two columns on the left are the hydrogen purification towers. The third and fourth are for flourine. The light gray inverted U's at the lower left are the hydrogen outlet lines from one of the cells. The gas exits from the towers are visible at the top, the small diameter tubing teed into these exit lines being used to carry away the hydrogen fluoride formed during regeneration.

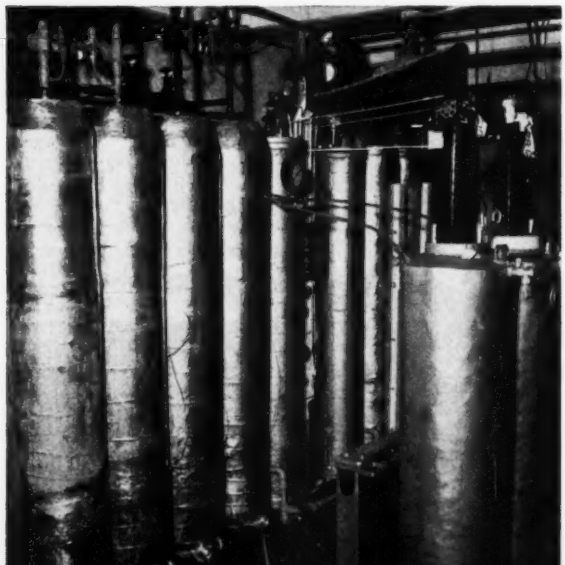


Figure 2. Fluorine Purification System (left) and Sulfur Hexafluoride Dryers and Condenser-Receivers (right) Mounted on Scales

To effect a more efficient removal of hydrogen fluoride, a three-tower system is used for each gas, with one tower on regeneration and two on stream (a partially spent tower backed up by a freshly regenerated tower).

Waste Fluorine Disposal. An effective fluorine disposal system is an essential part of any plant that manufactures or uses fluorine. The gas must not be vented to the atmosphere. The most convenient way of disposing of this gas is to burn it in the presence of city gas (natural or producer) and to absorb the hydrogen fluoride which is formed in an alkali solution (*16*).

In these operations, the waste gases that contained fluorine were piped to a specially designed nozzle located at the center of a ring burner supplied with city gas. When fluorine gas entered the combustion zone, the brilliance of the flame increased because of the vigorous reaction of fluorine with hydrogen and hydrocarbons. This phenomenon can therefore be used to indicate when the purging of the cell for maintenance shut-downs and of the purification towers, prior to their regeneration, is complete.

A funnel-shaped nickel or Monel hood placed over and around the burner collects the combustion gases which are drawn by means of a blower to a coke-packed scrubbing tower through which potassium hydroxide solution is circulating. Spent alkali liquor, in which the potassium hydroxide has been largely converted to potassium fluoride, is recausticized by treatment with lime to precipitate calcium fluoride and reform the alkali:

$$\text{On stream:} \quad KOH + HF \rightarrow KF + H_2O$$

$$\text{Regeneration:} \quad 2KF + Ca(OH)_2 \rightarrow 2KOH + CaF_2$$

The insoluble calcium fluoride is allowed to settle and the alkali liquor is decanted through a filter.

Packaging. The mechanical compression of fluorine has so far been successful only in producing pressures of the order of 30 p. s. i. (*10*). The resistance of most metals to attack by fluorine depends upon the formation of a protective film of metal fluoride which prevents further reaction; therefore, if this layer is ruptured or removed by flection or friction, which is almost unavoidable in mechanical pumps, attack on the metal will continue until the part fails. Another problem which has not yet been satisfactorily solved is the selection of an inert lubricant and/or pressure transmission fluid. Hence liquefaction and compression by evaporation have so far been the only successful means of packaging fluorine under pressures of several hundred pounds.

In our installation, as a precautionary measure, fluorine gas stripped of HF is passed through a nickel tube at 300°C. to decompose any explosive fluorine compounds that might be present (such compounds have been mentioned, particularly in the German literature). The gas is then cooled and led to a stainless steel condenser-receiver cooled by liquid nitrogen contained in a stainless steel Dewar flask which can be raised or lowered as required for condensation and evaporation. When sufficient liquid fluorine has accumulated, the Dewar flask is lowered and the receiver is heated to distill the fluorine through a $^1/_2$-in. nickel manifold into previously evacuated (1–5 mm. of Hg) steel cylinders to a pressure of 400 p. s. i. g. The condensation and cylinder filling operations are carried out behind a $^1/_2$-in. steel barrier through which the necessary operating controls extend (see Figure 3). After storage in a test room for at least 24 hours at 130°F., filled cylinders are reinspected for leakage.

Particularly when handling compressed fluorine it is imperative that the system be absolutely free of water, oil, grease, rust, scale, and metal filings. The heat of reaction of fluorine with any of these materials may be sufficient to ignite the metal piping. All cylinders are therefore given a thorough internal examination and pickled, if necessary, to remove rust. Valves are dismantled, thoroughly cleansed, repacked with solvent-washed packing, and pressure tested before each filling. Braided copper and Teflon (polytetrafluoroethylene) washers are used as packing.

Fluorine is marketed in cylinders of the sizes shown in the table below.

Hazards and Safety Measures. Animal experiments have shown elemental fluorine to have a toxicity definitely greater than and distinct from that of hydrogen fluoride (*17*). However, the intense characteristic odor of fluorine, which is detectable in trace amounts, affords excellent warning of leakages. Airline or oxygen masks will afford protection against fluorine-contaminated atmospheres.

No treatment has been definitely established for the severe inhalation of fluorine; but since its first physiological action is that of a strong pulmonary irritant, the established treatment for HF inhalation would seem logical. Details can be found in the Manufacturing Chemists' Safety Data Sheet SD-25 (*8*).

Flash burns of fluorine have been shown to be second-degree thermal burns. Their treatment is the same as for thermal burns. For more serious fluorine burns where the damage may be caused by both heat and fluoride ion, the physician may decide to follow the treatment for hydrogen fluoride injuries as also discussed in the Safety Data Sheet SD-25 (*8*).

Safety clothing gives very little protection against elemental fluorine. Therefore, the best defense against fluorine is frequent and painstaking inspection of all equipment and provision of adequate safety barriers and ventilation (*5*, *7*, *9*).

CHLORINE TRIFLUORIDE

Chlorine trifluoride, ClF_3, which melts at −83°C. and boils at 11.3°C., is a colorless gas which can be condensed to and maintained as a pale yellow liquid at 25°C. under a pressure of only about 15 p. s. i. g. (*3*). Chlorine trifluoride is almost as active chemically as fluorine itself. Because this compound contains 61.5 per cent fluorine and is almost as vigorous a fluorinating agent as elemental fluorine, and also because it is easily stored and shipped as a liquid under relatively low pressures, it is attracting interest as a possible substitute for fluorine for some of the uses of the latter. Solutions of chlorine trifluoride in anhydrous hydrogen fluoride, for example, have been found to fluorinate some organic compounds very smoothly without the disintegrating effect so often observed with elemental fluorine. It has also been reported that chlorine trifluoride was produced in Germany during World War II to be used in incendiary shells and jet propulsion fuels.

Figure 3. Filling Fluorine Cylinders behind Barrier

Chlorine trifluoride is produced when stoichiometric amounts of chlorine (1 volume) and fluorine (3 volumes)are mixed and heated at 300°C. Our studies on the production of chlorine trifluoride were on a smaller scale than those used for the other products discussed here and were actually large-scale laboratory operations. The output of a 250-ampere fluorine cell was led through a Monel preheater held at 290°C. and thence to an externally heated Monel reactor packed with nickel turnings. Before entering the reactor, the proper proportion of chlorine was bled into the fluorine stream from a cylinder, through a rotameter. The products of reaction were liquefied in a Monel spiral condenser cooled with a dry-ice, trichloroethylene mixture and collected in one of a pair of steel storage receivers. The principal impurities are chlorine monofluoride, and occasionally fluorine; these are substantially removed by maintaining the receiver at 0°C. (ice-brine bath) and allowing them to vaporize through a copper reflux condenser which is kept at −80°C. and hence retains the chlorine trifluoride.

When sufficient product has been collected in one receiver, the other is put on stream while the contents of the first are transferred to steel cylinders. To effect the transfer, the valve to the reactor is closed and heat is applied by means of a warm water jacket. When the pressure in the receiver reaches 10–15 p. s. i. g., the valve to the evacuated, chilled cylinder is opened to permit the liquid chlorine trifluoride to flow in. Shipments have been made under permit in ICC-3A-2015 steel cylinders following performance tests which indicated negligible corrosion after 30 days at 212°F. Chlorine trifluoride has been stored for six months at 130°F. in ICC-3A-480 steel cylinders with no corrosion, and permission to ship it in these lighter cylinders has been granted. The cylinders are equipped with a

	Cylinder characteristics					
ICC spec.	*Height, in.*	*Diameter, in.*	*Weight, lbs.*	*Capacity, cu. in.*	*Fluorine content, lbs.*	*Pressure, p. s. i. g.*
3A-2015	26.5	4.25	20	326	0.5	400
3A-2015	51	9.03	125	2640	4.0	400
3A-2300	58	10.75	229	4150	6.0	375

chlorine valve with braided copper and Teflon packing.

Because chlorine trifluoride approaches fluorine in chemical activity, the same rigorous precautions (*3*) are observed in the inspection and cleansing of the reactor, piping, valves, and cylinders. First aid treatment for burns is also the same.

SULFUR HEXAFLUORIDE

In contrast to chlorine trifluoride, sulfur hexafluoride (SF_6) is one of the most stable and chemically unreactive compounds known (*12, 15*). Sulfur hexafluoride melts at −50.8°C. under pressure, and sublimes to a colorless gas at about −63.7°C. Because of its chemical inertness and excellent dielectric properties, sulfur hexafluoride is receiving attention as an electrical insulating gas for high-voltage equipment.

Sulfur hexafluoride is produced by the direct combination of sulfur and fluorine. In our pilot plant, this reaction was carried out in a horizontal steel reactor, 12 in. in diameter and 30 in. long. The reactor is divided into a charging chamber and a combustion chamber by means of a baffle which extends to within a short distance of the bottom so that the charge of molten sulfur forms a liquid seal between the two chambers. The charging chamber is equipped with a port for the periodic introduction of sulfur, and is electrically heated and lagged. The combustion chamber is provided with a hand-port (for cleaning out), a thermocouple well, and a water-jacketed steel fluorine injection nozzle. The reaction is exothermic, and natural conduction and convection suffice to keep the temperature of the sulfur in the reaction zone at 138–149°C. when charging 1.5 lb. of fluorine per hour. At higher fluorine feed rates, *e. g.*, 3 lb. per hour, external cooling is required.

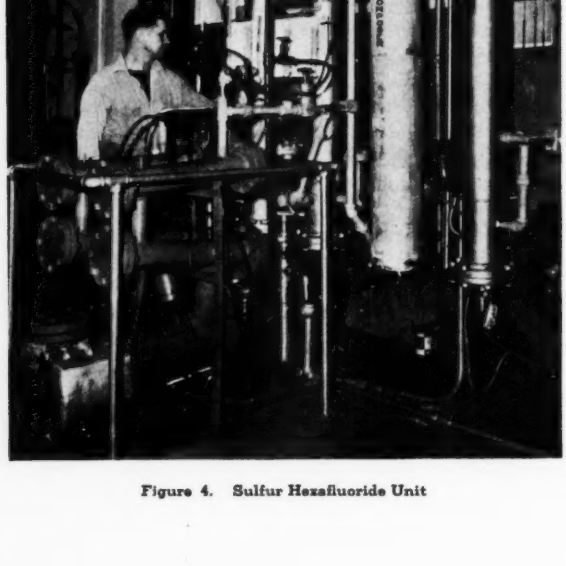


Figure 4. Sulfur Hexafluoride Unit

In Figure 4, the sulfur hexafluoride generator appears at the lower left with the lagged charging chamber to the left. Above the combustion chamber are two parallel sets of condenser settlers which remove distilled sulfur. The five towers, from left to right, are the two primary alkali scrubbers, the decomposer, a cooler (which was later converted to a scrubbing tower), and part of the first of the two final scrubbers.

The major impurities in the crude gas leaving the combustion chamber are sulfur dioxide, excess fluorine, hydrogen fluoride, sulfur monofluoride (S_2F_2), sulfur tetrafluoride (SF_4), disulfur decafluoride (S_2F_{10}), and nitrogen. The first five named are readily removed by an alkali scrubber, the S_2F_{10} can be decomposed thermally into sulfur hexafluoride and sulfur tetrafluoride (which is then removed in a second alkali scrubber), and the nitrogen is vented when the product is liquefied. Sulfur hexafluoride is nontoxic. On the other hand, the lower fluorides are very poisonous, hence their removal from the product is essential.

The first alkali scrubbing is accomplished with the alternate use of two 6-ft. steel towers, packed with steel Raschig rings, through which 20 per cent potassium hydroxide solution is circulated. The alkali solution enters the tower a short distance below the top so that the packing above the inlet serves to remove spray which would otherwise deposit solids in the decomposer inlet nozzle. When the alkali solution is spent it is regenerated with lime as described earlier in this article. The gas from the first scrubber passes through a 6-ft. section of 2.5-in. nickel pipe packed with nickel turnings maintained at 600°F. to decompose the S_2F_{10}, and thence to two more alkali scrubbers, in series, which complete the purification train. The purified gas is collected in standard inverted bell-type gas holders over 23 per cent brine.

A typical analysis of crude and purified sulfur hexafluoride is:

Component	*Crude gas, %*	*Dry purified gas*
SF_6	94.3	99.8
S_2F_{10}		Nil
$SF_4 + S_2F_2$	1.9	8 p. p. m.
SO_2	2.4	Nil
HF	0.4	Nil
Inerts	1.0	0.2%
Moisture	Trace	15 p. p. m.

For packaging in cylinders the sulfur hexafluoride is withdrawn from the holders by a single stage compressor which delivers the gas under a pressure of 100–150 p. s. i. g. through cooling towers and an adsorbent dryer to a condenser-receiver, a 6100-cu. in. lagged steel cylinder which is refrigerated by means of Freon-filled coils (Figure 2). By venting the receiver while under refrigeration, the nitrogen is expelled with some

loss of product. Liquid sulfur hexafluoride is then forced to flow into previously evacuated ICC-3A-2015 or 3A-2300 steel cylinders holding 3, 10, 100, or 150 lb. of product at 275–300 p. s. i. g.

METALLIC POLYFLUORIDES

The metallic polyfluorides are of potential industrial interest because they afford a readily controllable means of fluorinating organic compounds, for example, to make the chemically-inert non-inflammable fluorocarbons (*1*). Of this group, cobalt trifluoride is perhaps of greatest importance, but silver difluoride, manganese trifluoride, and mercuric fluoride have also received some attention. These compounds are made by the action of elemental fluorine on the oxides or salts of the metals. Thus,

$$2Co_3O_4 + 9F_2 \rightarrow 6CoF_3 + 4O_2$$
$$2AgCl + 2F_2 \rightarrow 2AgF_2 + Cl_2$$
$$2MnO_2 + 3F_2 \rightarrow 2MnF_3 + 2O_2$$
$$HgCl_2 + F_2 \rightarrow HgF_2 + Cl_2$$

The horizontal rotary kiln in which these preparations were made was fabricated from a 10-ft. section of 6-in. steel pipe, equipped with lifting flights and mounted on trunnions on an adjustable frame to permit varying the slope of the kiln and consequently the residence time of the solid reactants. A variable speed drive is used to adjust the rotational speed in the range 5–14 r. p. m. Five sampling ports, distributed along the length of the kiln, and an exploring thermocouple are also provided. Solid reactant is admitted from a feed hopper at one end of the kiln by means of a variable speed screw conveyor to permit changes in the charging rate. Fluorine is admitted at the opposite end. Excess fluorine, along with gaseous reaction by-products, leaves the reactor through an electrostatic precipitator, which returns dust to the kiln, and thence to the fluorine disposal burner. A product receiver is connected to the kiln through a 4-in. butterfly valve to permit changing the receiver without interrupting production. The kiln is equipped with two glands packed with lead foil covered asbestos backed by graphitized (unoiled) asbestos. Through a lantern ring the glands are swept with nitrogen to prevent attack on the packing. This arrangement was found to be satisfactory.

Figure 5 is a general view of the polyfluoride reactor with the charging end at the left and the receiver at the lower right. The gas main feeding the burners and an asbestos board hood are also shown. On the control board behind the reactors are the manometers and sight bubblers necessary for the control of the process.

Cobalt trifluoride and manganese trifluoride were produced as free-flowing powders at rates of 2.7 to 5.2 lb. per hour using from 10 to 40 per cent excess fluorine and reaction initiating temperatures of 430 to 600°F.

The kiln is also adaptable to batch production by operating horizontally and placing a retaining dam at the product outlet end. Silver difluoride and mercuric fluoride were made in this way in batches up to 40 per cent of the kiln volume with reaction-initiating temperatures of 100 to 400°F. These fluorides may also be made, of course, by continuous operation.

The products were packaged in tin-plated steel cans having outside lead-soldered seams and friction or screw cap lids.

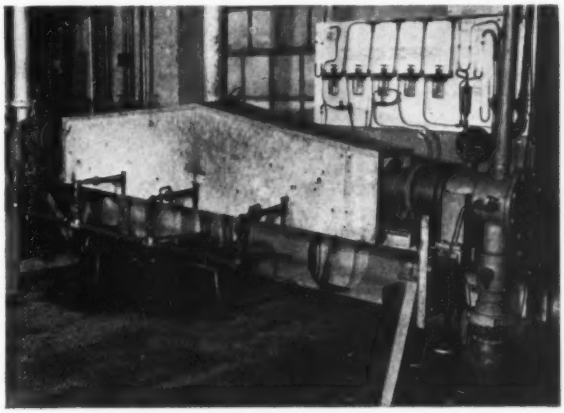


Figure 5. Reactor for Metallic Polyfluoride Production

CONCLUSIONS

During many months of operation of the fluorine cells and the auxiliary equipment for the production of chlorine trifluoride, sulfur hexafluoride, and the metallic fluorides, only one injury was sustained by the personnel engaged in this work. This injury involved the handling of anhydrous HF and was occasioned by a disregard of safety rules. Experience with this plant has taught where danger may exist and how those dangers can be avoided. With that knowledge and with a strict observance of a fluorine safety code, fluorine, which in the past has justifiably been feared, can be handled without serious industrial hazard.

There can be little question that fluorine and its compounds will play an increasingly important role in industry. The growing assurance that the major technical difficulties have been solved, and the knowledge that fluorine and many of the laboratory fluorine chemicals have already reached the stage of semi-commercial scale production will very probably overcome the hesitancy of many to consider the use of these materials in existing and new operations.

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AN EXPERIMENT FOR THE pH METER

ROBERT L. PECSOK
University of California, Los Angeles, California

THE present trend for course material in quantitative analysis is to reduce the number of time-consuming, classical procedures to a minimum, retaining only a sufficient number to introduce the student to careful techniques. The increasing scope of analytical chemistry and the increasing use of analytical instruments in industrial laboratories require us to reconsider carefully the content of our courses.

The introduction of instrumental methods to sophomore courses has not found universal acceptance; however, electrolytic determinations have been standard for many years and are discussed in considerable detail in the popular textbooks. Colorimetric determinations are now a part of most elementary courses; the instrument may be as simple as Nessler tubes, but more probably a Duboscq or photoelectric colorimeter.

The basic principles of a pH meter are covered in the theoretical treatment of oxidation-reduction chemistry of most courses. In view of the importance and frequency of the determination of hydrogen ion concentration we feel that the use of the pH meter has a definite place in elementary courses. The cost of such a program is not unreasonable. Our investment in pH meters is less than that in platinum electrodes for electrogravimetric work.

Although acidimetry is part of our first semester's work we postpone the pH meter until the second semester, partly because of the fewer students involved, and partly because there are several experiments which must be staggered due to the apparatus involved. In this manner we are able to keep our equipment in use nearly constantly, and six meters are ample for a class of fifty.

Few of the standard texts discuss the pH meter, and almost none give detailed experimental procedures. Since inevitably some students must do the laboratory work before it can be discussed in lecture, they are provided with a summary of the principles involved in the construction and operation of the meter. This includes a discussion of the electrode system with equations for the potentials of both electrodes. It is shown that the equation relating potential to pH is linear and that therefore a single scale on a calibrated and properly standardized voltmeter can be read either in volts or directly in pH units. The amplifier and voltmeter are treated as a unit with the details of the circuits omitted. The advantages of a pH meter are stressed, particularly its accuracy, convenience, and adaptability to applications involving recording and control.

In the selection of experimental work, we imposed these requirements: (1) experimental data that could be compared to theoretical data calculated by principles discussed in class, (2) determinations that would be more difficult by other methods, and (3) an unknown, the results of which could be graded. The titration of a polyprotic acid and its salts fulfills these conditions.

The procedures which follow were designed for use with a Beckman Model H-2 line operated pH meter. They are taken in part from the instructions furnished by the manufacturer.[1] With minor modifications in the standardization and reading, they can be used for any of the commercial instruments available. They are obviously designed to keep breakage and other troubles at a minimum. In four semesters' experience with about 175 students our only maintenance has been replacing potassium chloride in calomel electrodes and one broken glass electrode. More rugged electrodes have recently become available.

[1] Bulletin 190-B, Beckman Instruments, Inc., South Pasadena, California.

PROCEDURES

Care of Electrodes. The electrodes are fragile and easily ruined if scratched or bumped. Never touch the glass parts of the electrodes with anything. The electrodes are always stored in distilled water. When transporting the instrument, raise the electrodes and turn them toward the meter.

Procedure for Standardization.

1. Immerse the electrodes in a beaker of distilled water.
2. Set range switch to "start."
3. Connect power line plug to 110-V. a.-c. outlet.
4. Allow a five-minute warm-up period.
5. During warm-up period prepare standard buffer by either of the following methods.
   (*a*) Dilute 5 ml. of Beckman concentrated buffer solution to 125 ml. using a volumetric flask. Note the temperature of the buffer, and its correct pH from the bottle label.
   (*b*) Prepare a saturated solution of potassium acid tartrate (solubility—0.6 g./100 ml.) The pH of this solution is 3.57 with a negligible temperature coefficient.[2]
6. Meter needle should read exactly 7.00 pH. If not, adjust with small screw above needle pivot.
7. Set "temp" control to temperature of buffer.
8. Set range control to "neut" position. The range switch should always be snapped smartly from one position to another and not stopped at an intermediate point.
9. Rinse and fill beaker with buffer solution. Immerse electrodes.
10. Set range switch to 0–8 pH range.
11. Adjust the meter needle to read the pH of the buffer by turning the "standardization" control.
12. Set range control to "neut" position. Set adjustable pointer to indicate position of meter needle. Pointer is controlled by small knob at lower left of meter panel.
13. The coincidence of the pointer and the needle with the range switch in "neut" position should be checked frequently as this indicates any drift of the amplifier. Adjust the meter needle if necessary with the "standardization" control.
14. Always leave the range switch in the "neut" position between readings or when changing solutions, and whenever the electrodes are not immersed. Failure to observe this precaution will cause polarization of the glass electrode. The polarization is temporary but causes readings to drift for several minutes after reimmersion.
15. When changing from one solution to another, raise the electrodes and rinse them with a stream of water from a wash bottle. (For student use, it is preferable not to dry them unless the solution is poorly buffered. In such cases, soft absorbent tissue may be used.)

Measurement of pH.

1. With the range switch in the "neut" position, immerse electrode in sample.
2. Check coincidence of pointer and needle and adjust if necessary.
3. Switch range control to proper pH range.
4. The needle now indicates the pH.
5. Return range switch to "neut" position before raising electrodes.

Titration Curve of Phosphoric Acid.

1. Prepare a liter of 0.2 *N* sodium hydroxide (carbonate-free).
2. Pipet a 25-ml. portion of approximately 0.1 *M* phosphoric acid into a 250-ml. beaker.
3. Immerse electrodes in the acid, dilute with water if necessary to cover the bulb of the glass electrode (see the figure).

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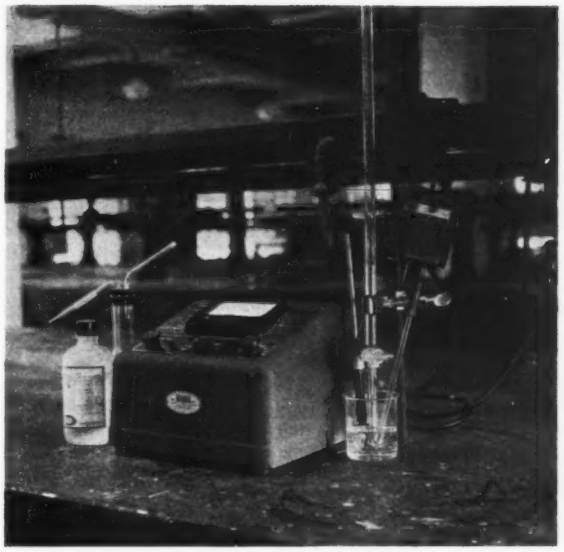

Experimental Set-up for Titrations

4. Arrange stirrer so that there is no danger of contact with the electrodes. The solution should be stirred continuously and the electrodes left immersed during the entire titration. It may be necessary to ground the meter to prevent drifting of the needle due to electrical pick-up of the amplifier.
5. Mount the buret and commence the titration with sodium hydroxide solution. Measure and record the pH after each increment of base. Take sufficient points so that an accurate titration curve can be plotted, *e. g.*, every 3 to 5 ml. between end points and every 0.5 ml. or less near the end points. Do not continue the titration beyond pH 11 to 12. Above pH 11 significant corrections are necessary. At very low hydrogen ion concentrations the glass becomes sensitive to sodium ion as well as hydrogen ion.
6. Plot the curve on graph paper. Plot the theoretical curve and compare, particularly, the pH at the equivalence points. Note the pH for the first two equivalence points of the experimental curve and use these values in subsequent titrations.

Standardization of Base.

1. Obtain a sample of standard sulfuric acid from stockroom.
2. Pipet a 25-ml. portion of acid into the beaker and titrate with base, following change of pH. The student should add a drop or two of a suitable indicator and follow its color change. Obtain a sufficient number of points to plot a titration curve. Plot this curve on the same graph as the phosphoric acid curve and compare the two.
3. Calculate two normalities for the base, using the end points determined from the phosphoric acid curve. The normalities will differ depending on the amount of carbonate in the base.

Analysis of Mixed Phosphate Solutions.

1. Two separate samples are to be analyzed.[3] Take a 25-ml. sample and titrate to both end points (proper pH has been determined above) with standard acid or base as required. Intermediate readings need not be taken except as a guide to the approach of an end point.
2. Identify the components of each unknown mixture and report the concentration of each.

[3] Various mixtures of H_3PO_4-NaH_2PO_4 and NaH_2PO_4-Na_2HPO_4 can be used.

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J. G. TOLPIN, J. DANACZKO, JR., R. A. LIEWALD, E. A. MAYERLE, E. H. MAYERLE, E. B. OLSZANSKI, V. C. SEKERA, and C. ZIMMER

The University College, Northwestern University, Chicago, Illinois

THE interrelationship of the work of the Russian scientist with that of scientists in other countries receives attention as part of the effort to develop a correct appraisal of the importance of Russian scientific contributions and of the importance of studying the Russian scientific literature in the original. As one aspect of this study, a group of chemists and chemical bibliographers studying the Russian language in a course for scientists, taught by the senior author at Northwestern University, analyzed the references cited by authors of articles on organic chemistry published in seven Russian journals during six years. This was thought to be a suitable method; we agree with those who maintain that the usefulness of the chemical literature in any given language is more correctly indicated by the extent to which it is cited by chemists rather than by its absolute proportion in the world's literature of this field.

The following years were selected for the study under report: 1913, for the most suitable representation of Russian chemistry before World War I; 1929, representing the year when the first Five Year Industrialization Plan of Russia got under way; 1939, as representative of pre-World War II chemistry; and 1946, 1947, and 1948, as representative of the post-World War II development of chemistry.

A specialized technique for uniform collection and classification of the data was developed and the cards reporting these data were gathered by R. A. Liewald, the coordinator of this project, who was also responsible for tallying the data. Altogether 17,368 references, comprising 16,255 references to journal articles and a few patents, and 1113 monographs and books, etc., were classified.

We established that the Russian chemist relies on the chemical literature of other countries to a greater extent than the American chemist. The German chemical literature was until recently the principal source of his information, as far as organic chemistry is concerned. This factor was especially pronounced at the time of rapid expansion of research (1929). His ability to rely on his own literature has been steadily increasing percentage-wise through the years. The increase and decrease in his use of the chemical literature of the individual countries is based primarily on the significance of these countries as contributors to chemical research. For summarized information required for industrial expansion he makes much use of the monographic and review literature in chemistry. A considerable proportion of that type of literature is prepared for him in the Russian language, but foreign information figures high in these reviews, as indicated by larger percentages of American references in the review literature.

The references studied came from the journals listed in Table 1.

TABLE 1
Russian Journals Surveyed

Year	*Journal*		*Number of articles*
1913	J. Russ. Phys.-Chem. Soc.		71
1929	J. Russ. Phys.-Chem. Soc.		95
1939	J. Gen. Chem. (Zhurnal Obshcheĭ Khimii)	237	
	J. Appl. Chem. (Zhurnal Prikladnoĭ Khimii)	98	
	Reports Acad. Sci. USSR (Doklady Akademii Nauk)	29	410
	Progress of Chem. (Uspekhi Khimii)	11	
	Org. Chem. Ind. (Promyshlennost' Organicheskoĭ Khimii)	35	
1946	J. Gen. Chem. (Zhurnal Obshcheĭ Khimii)		106
1947	J. Gen. Chem. (Zhurnal Obshcheĭ Khimii)	202	
	J. Appl. Chem. (Zhurnal Prikladnoĭ Khimii)	51	
	Bull. Acad. Sci., Dept. Chem. Sci. (Izvestiya Akademii Nauk, Otdelenie Khimicheskikh Nauk)	52	316
	Progress of Chem. (Uspekhi Khimii)	11	
1948	J. Gen. Chem. (Zhurnal Obshcheĭ Khimii)		157
	Total		1155

The different types of articles were distributed as follows:

Articles reporting experimental work	1038
Theoretical articles	36
Review articles	63
Not classified	18

For the years 1946 and 1948 only the most significant journal, *Journal of General Chemistry* (*Zhurnal Obshchei Khimii*) was studied in order to determine more clearly the present-day trends. This journal or its predecessor, *Journal of the Russian Physico-Chemical Society* (*Zhurnal Russkogo Fiziko-Khimicheskogo Obshchestva*) was sur-

[1] Presented before the Division of Chemical Literature and the Division of History of Chemistry at the 118th Meeting of the American Chemical Society, Chicago, September 7, 1950.

TABLE 2
Russian Contributions to Chemistry, Per Cent of World's Total

	1909	*1910*	*1913*	*1918*	*1919*	*1929*	*1930*	*1939*	*1940*	*1941*[a]	*1947*
Entire field of chemistry. Source: E. J. Crane (*1*)	1.2	...	2.5	0.7	...	3.4	...	11.1	14.1	...	8.2
Inorganic, physical, and mineralogical chemistry. Source: I. I. Zaslavskiĭ (*9*)	...	6.9	7.7	...	0.6	...	8.2	15.0	17.3	20.6	...

[a] For the first 23 weeks of 1941.

veyed for all the years studied, since it was shown by Paul W. Howerton (*4*) that this journal is the major vehicle of organic chemical papers in Russia.

We have attempted to survey as much of the Russian organic chemical literature for the years indicated as would be in accord with over-all growth of the Russian contributions to the field of organic chemistry. It must be pointed out here that our selection of organic chemistry was based exclusively on the desire to cover one field of chemistry of importance in the professional work of some of the members of this group; to cover a wider field would have constituted a larger task than that possible for us at this time. However, we have exhausted the main sources of Russian organic literature available to us without reaching the proportions for 1939 and 1947, as would be indicated by the increase of Russian contributions shown by *Chemical Abstracts*, if we use the entire 1913 number of references (938) as a basis (*1*). If we chose inorganic or physical chemistry as a gage of the progress of Russian contributions we would have to cover a still larger amount of material. This is shown by data for Russian publications in the fields of general, inorganic, physical, and mineralogical chemistry during the years 1875 to 1948 reported by I. I. Zaslavskiĭ (*9*), and based on an examination of the abstracts in *Chemiches Zentralblatt*. He credited the Russian chemists with their works published both in Russian and in non-Russian journals. An excerpt from Zaslavskiĭ's data is compared in Table 2 with some data based on *Chemical Abstracts*. According to Zaslavskiĭ, physical chemistry showed a more rapid growth during the period surveyed than other branches of chemistry in Russia, just as anywhere else. However, of the individual subdivisions of physical chemistry, structure of matter was the favorite subject outside of Russia, while in Russia researches on electrochemistry and thermochemistry received just as much attention, and colloid chemistry was treated in about half as many publications as the former.

Discussions based on the specific questions we attempted to answer and conclusions drawn from our study follow:

(1) Does the Russian organic chemist rely on his own literature to the extent to which it is represented in the world's literature? The data of E. J. Crane and of I. I. Zaslavskiĭ, although they refer to the entire chemical literature in the first case and to some branches of chemistry not covered by us in the second case, can be used to answer this question. When correlated with our data, shown in Table 3, they indicate that he relies on it to a greater extent, which is natural for him, just as it is for American chemists to quote their own literature more than others, as shown by Sheppard (*6*) and by Fussler (*2*).

An analysis of the references used by the authors publishing in the Russian *Journal of General Chemistry* (*Zhurnal Obshcheĭ Khimii*) for 1939 (total number of references, 4190; number of journal references, 89.3 per cent, or 3741) was published by A. F. Platé in *Progress of Chemistry* (*Uspekhi Khimii*) (*5*). The data for journal references given by Platé are quoted in Table 3 for the sake of comparison. His distribution is not far from ours. Platé's classification is based on language; the sum of the U. S. and British publications also includes a part of the publications using the three principal languages (English, German, and French). A redistribution of these was made by us to fit our classification.

TABLE 3
Distribution of References in the Russian Journals

Year	*Number of references*	*Per cent*					
		Russia	*Germany*	*United States*	*England*	*France*	*Other*
1913	938	29.8	49.9	1.5	7.0	8.2	3.6
1929	1358	19.4	59.0	4.5	7.4	8.0	1.7
1939 (This study)	5770	26.8	40.8	12.9	6.9	6.4	6.2
1939 (Platé)[a]	3741	26.1	45.3	18.6		8.9	1.1
1946	1490	28.4	39.6	16.9	7.6	4.7	2.8
1947	5645	32.6	30.6	19.6	7.2	5.2	4.8
1948	2167	37.9	35.8	12.7	5.9	4.1	3.6

Total references examined in this study, 17,368.

[a] Platé treated the references of one journal for 1939 only. He classified them according to the language, not the countries of origin.

The distribution of patents was observed by Platé to follow generally the same distributions as journal articles, which also supports our classifying these two types of references together.

It is seen from Table 3 that at no time did the Russian organic chemists quote their own literature during the period under study to an extent exceeding the maximum of 38 per cent of the world's chemical literature, which was reached in 1948, against 19 per cent at the beginning of the industrialization period. This fluctuation is not great, considering the special circumstances and the expansion of the Russian scientific publication activity. The sequence of the numbers of references in the various languages does not necessarily agree with the sequence of the numbers of publications cited. For instance, according to Platé the Russian authors quoted (in 1939) 60 German and 76 Russian journals, but the number of references to the German journals was much greater than that to the Russian.

(2) The decline in the proportion of German references is significant. The influence of German science on Russian research in organic chemistry went down from 59 per cent at the beginning of the industrialization to 30–36 per cent for the present time. The Russian chemist now uses his own literature at least as much as the German. This observation parallels those made by H. H. Fussler and by O. E. Sheppard for the influence of the German chemistry on the work of American chemists. Sheppard's data for references in the *Journal of the American Chemical Society* for the year 1933 and Fussler's data for percentage distribution of serial references in American journals on chemistry are quoted in Table 4.

TABLE 4
Distribution of References in the American Chemical Literature

	1899[a]	*1919*[a]	*1933*[b]	*1939*[a]	*1946*[a]
United States	25.1	38.2	46.6	48.5	53.1
Great Britain	12.6	9.9		16.0	12.7
Germany	49.1	40.5	39.3	25.0	24.0
France	9.9	7.8	8.3	3.0	4.8
Others and undetermined	3.3	3.6	5.6	7.5	5.4

[a] Data of H. H. Fussler (*2*).
[b] Data of O. E. Sheppard (*6*). Unlike Fussler's data, these percentages are based on languages, not on countries. His figure for publications in English-French-German (1.5%) was redistributed and included in the three respective figures.

Fussler states that "the national rank orders of numbers of citations to monographic material and serial material are in most instances the same."

Thus, Germany was the leading source of chemical information in the United States at least up to 1919 and in Russia up to 1946. Incidentally, according to Paul Walden[3], the German Chemical Society in 1911 had 3202 members against 6091 members of the American Chemical Society.

It is likely that Germany occupies the second place also in the British chemical literature (*6*), but a limited examination of the references in *Berichte* made by Sheppard showed that the German references amounted there to 90% of all references. This attitude of the German chemist to chemical research outside Germany is probably in existence also today (*10*).

(3) Germany lost its position as the leader in chemical information in both the United States (Table 4) and Russia (Table 3) as a result of the rise of American chemical research. Our data reveal that the influence of the United States chemical literature in Russian organic chemistry rose from 1.5 per cent in 1913 to 13–20 per cent at the present time. In Russia, but not in the United States, the increase of the volume of Russian scientific information also played a part. Sheppard's analysis showed that in the *Journal of the American Chemical Society* for 1933 only 0.7 per cent of the references were Russian. According to Fussler, Russia received the highest number of citations in the American chemical literature in 1946, the last year covered by his study. That number amounted to 1.97 per cent; in the same year, the Russian organic chemists cited American works to the extent of 16.9 per cent. Undoubtedly the language difficulty is not the only factor in the neglect of the Russian chemical literature in this country. Despite the successful efforts of *Chemical Abstracts* to represent it, the number of American chemists studying the Russian language and following the Russian chemical literature is as yet very small. A discussion of this subject was previously given by one of us in greater detail (*7*).

(4) Review articles in *Progress of Chemistry* (*Uspekhi Khimii*) as well as articles in *Industrial Organic Chemistry* (*Promyshlennost' Organicheskoĭ Khimii*) in 1939 and 1947 carry American references in greater proportion than Russian references, Germany retaining the place discussed above for the literature as a whole. This is in contrast to the material published in *Journal of General Chemistry* (*Zhurnal Obshcheĭ Khimii*) as seen from Table 5.

It may be suggested on the basis of these data that in surveying a field preparatory to his own experimental study of it, and in his work in the field of industrial organic chemistry, the Russian chemist finds more pertinent information in the American than in his own literature. However, more study of the current Russian chemical literature is needed for such an assertion on this point. Incidentally, in previous years the Russian chemical journal *Progress of Chemistry* (*Uspekhi Khimii*) used to carry a substantial number of reviews

TABLE 5
Distribution of References in Russian Reviews versus Articles on Experimental Work

		Ind. Org. Chem.	*Prog. of Chem.*	*J. Gen. Chem.*
1939	Russia	131	39	825
	United States	178	89	325
	Germany	339	176	1178
1947	Russia	...	116	1185
	United States	...	422	405
	Germany	...	392	954

[3] Quoted from I. I. Zaslavskiĭ (*9*).

translated from *Chemical Reviews* and similar journals. A series of books was recently announced (*8*) which will be published under the general title "Chemistry and Chemical Technology" ("Khimiya i Khimicheskaya Tekhnologiya"). It will be sponsored by the Ministry of the Chemical Industry of the U. S. S. R. and will review in form of abstracts the progress of chemistry outside Russia.

The distribution of the references to books and handbooks is compared below with that given by A. F. Platé for 1939. He recorded a total of 322 books compared with a total of 396 books for the same year recorded by us for organic chemical articles in the various journals. The trend is seen to be generally similar to that of the periodicals, but the increase of the American references is more rapid, in accordance with the discussion above.

TABLE 6
Distribution of the Russian References to Monographic Literature, Per Cent

Monographs and handbooks quoted	*Russia*	*Germany*	*United States*	*Britain*	*Others*
1913 54	37.0	51.9	0	0	11.1
1929 114	25.4	68.4	0.9	1.8	3.5
1939 332 (Platé)[a]	66.2	26.8	5.7		1.2[b]
1939 396 (This study)	71.2	18.7	5.1	4.0	1.0
1946 100	71.0	18.0	9.0	0	3.0
1947 354	57.9	20.6	16.7	1.4	3.7
1948 95	66.3	10.5	20.0	2.1	1.1

Total 1113 monographs covered by this study.

[a] Distribution according to languages and not countries.
[b] French only.

(5) The institutions at which the research reported was done are classified in Table 7.

TABLE 7
Source of Russian Articles Analyzed, Per Cent

	1913	*1929*	*1939*	*1946*	*1947*	*1948*
Schools	73.2	52.6	19.1	12.3	26.1	21.5
Research Institutes	18.3	30.5	63.5	73.5	59.4	71.8
Not given	8.5	16.9	17.4	14.2	14.5	6.7

As could well be expected, universities, which occupy the leading position in chemical research in 1913, are gradually giving up that position to research institutes, a good proportion of which serve industry and percentage-wise have almost exchanged places with the universities by 1946.

(6) The availability of the Russian scientific literature in this country is an important and complicated question. We have attempted to determine only the availability of the journals referred to, leaving out of account the monographic literature. Russian patents were considered as available if issued before 1930. A list of available journals was prepared based on regular coverage of these journals in *Chemical Abstracts* and the availability of the journal to subscribers in this country, as indicated by the official list (*Periodica U. S. S. R.*) published in Moscow by "International Book" (Mezhdunarodnaya Kniga) for the respective years. Thus, our figures for the availability actually show the percentages of the serial reference literature that *could have been* available to us in the years of publication, had our libraries subscribed to them, but do not indicate their actual availability. Furthermore, those journals which are actually available may be found in some cases only in very few American libraries. There is no complete list of Russian chemical publications available in this country, but we have a good chance of obtaining a photostat through the American Chemical Society photocopy service if a given publication can be found in one of the major libraries. Nevertheless, the figure for the potential availability of the Russian periodical reference literature is surprisingly high and exceeds 80 per cent as a rule. The availability percentage would, of course, be much lower if we were to make a survey of the references cited in the Russian monographic literature, for Russian chemists are likely to refer there more frequently to various provincial publications, bulletins, reports of research institutes, etc., *i. e.*, literature which is being freely circulated in Russia, but is of a type that would be classified in this country as private reports of industrial organizations and would receive only limited circulation. On the other hand, a part of that literature exists in U. S. S. R. only because of the limitations of the established chemical periodicals. At least that was the situation in 1939, according to A. F. Platé (*5*). Certain industrial publications are now withheld from circulation outside of Russia, in all likelihood for security purposes.

(7) It is noteworthy that the chemical literatures of England, France, and of the minor contributors did not undergo any drastic changes in their representation in the reference literature under discussion.

(8) If the list of Russian journals most frequently referred to is supplemented by the most frequently mentioned non-Russian journals a final list is obtained which does not differ very much from the list of journals most frequently cited by American chemists (*6, 2, 3*), with the exception that Russian journals occupy on this list a place close to the top and abstracting journals are also prominent. A. F. Platé's analysis (*5*) of the references in the 1939 volume of *Journal of General Chemistry* (*Zhurnal Obshcheĭ Khimii*) actually gave this result when compared with O. E. Sheppard's list based on the references in the 1933 volume of the *Journal of the American Chemical Society*. There is a list of some 35 prominent chemical journals in the world, regardless of the language, and every analysis of references finds more than half a dozen top journals. The composition of the top group changes, however, from country to country and also with the field of chemistry.

We did not study the date distribution of the references in the Russian organic chemical literature of recent years, but it is the impression of one of us (J. G. T.) that we would probably find a larger percentage of older references being quoted than in the American

chemical literature; the Russian chemist is trying to absorb the experience of the past generations of chemists in an attempt to establish his own way in many divisions of the field of chemical research.

ACKNOWLEDGMENT

We wish to extend our thanks to the library of Universal Oil Products Company, Riverside, Illinois, for the loan of many of the Russian journals used in this study.

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AN ELECTRIC FLASK HEATER

FREDERICK MATHEWS
Beloit College, Beloit, Wisconsin

The purpose of this paper is to describe the construction of a very inexpensive but extremely reliable and convenient electrical flask heater, made from common and readily obtainable materials. The original idea of this heater is not the author's, and this article is merely a complete description of a composite, with refinements, of several types observed in various laboratories in the last ten years. The actual construction of the heater demands no mechanical skill or special tools, and, in fact, most of the heaters in our laboratory were made with student help or were made entirely by students. This type of heater can be used only in conjunction with a variable-power transformer.

Figure 1

The flask has just been forced into the depression for the first time. Note that the emergent wire is coiled and not straight.

The performance of these heaters has been extremely satisfactory in every way. They are safe, durable, and very flexible in all applications, and with reasonable care they will last for years. Temperatures obtainable with liquids in the flask can be varied from 40° to 400°C. or more depending upon the size of the heater.

The materials for the construction of a 300-ml. flask heater include a one-pound coffee can with two 1/4-in. holes drilled, glass insulators, coiled resistance wire (22 ft. of B&S Nichrome or chromela wire), and sufficient asbestos cement (Johns-Manville No. 352).[1] The mandrel upon which the resistance wire is coiled is made of a piece of heavy wire about 18 in. long and 1/8 in. or slightly more in diameter with one end bent in the form of a crank handle. A small hole will serve to fix the end of the resistance wire during the winding. The wire coil is slipped off the mandrel and then pulled out to about 3 1/4 ft. in length. Straighten the ends for about 5 in. The volume of dry cement needed is difficult to estimate but usually a volume equal to about three or four containers will suffice. Add water to the cement with kneading until the cement reaches a putty-like consistency; when no water can be brought to the surface of it when it is squeezed hard, enough water has been added.

[1] Johns-Manville Corporation, 22 East 40th St., New York, can supply the address of the nearest dealer. Local heating-equipment concerns can supply asbestos cement that will often be suitable for use in heaters.

Figure 2

The coil has been forced into the side of the cavity and has been partly covered with cement.

Insert one glass insulator (a glass tube $^1/_2$ in. in length with a flange at one end) in the lower hole of the can and spread an inch layer of cement, working it well into the corners and around the glass insulator. Put the resistance wire in place, insert the other glass insulator, and place cement all around the sides of the heater, retaining a depression about the shape of the flask bottom. Build up to within about $^1/_2$ in. of the top of the container. When the cavity is just slightly smaller than the size of the flask force the flask into the cavity (see Figure 1).

The resistance wire is now pushed into the cavity side (Figure 2). The wire will not stay in place very well at this stage until it is covered with fresh cement. To do this, take a small amount of the cement and force it into the coiled wire with a finger. The covered coiled wire should just faintly show (Figure 3).

The heater can be dried with the flask in place. This will prevent cavity deformation due to the shrinkage of the cement. Heating at 100° to 120°C. overnight will drive off enough water to permit further drying with the flask removed. Electricity should not be passed through the wire until the cement is quite dry, because of possible wire corrosion by the wet cement. After the heater appears to be dry pass 40 to 70 volts (depending on the size of the heater) for several hours with no flask to drive out any residual moisture. The heater is then ready for use.

The considerable shrinkage that is noted with some cements is not undesirable for it permits more rapid drying at the edge of the heating block. However, a little fresh cement should be forced down along the sides when the cement of the heater is dry. This will take out the looseness between the heating block and the container. Cracks, if any develop, can be patched in this manner.

The wires emerging from the insulators must be fixed so they can easily be connected to the power supply and yet will not break off with heavy use. To do this, cut the wire so only $^7/_8$ in. protrudes from the end of the glass insulator. With long-nose pliers bend the end around and force about $^1/_4$ in. of the end back into the glass tube. This will form a strong $^1/_4$-in. loop that can easily be clamped with the small battery-clamp connectors, but cannot be broken off. The small $1^1/_2$-in. battery clips of the "Pee-Wee" type, when covered with a 2-in. length of $^3/_8$-in. rubber tubing, make safe and convenient connectors. A heat-resistant aluminum paint applied before or after fabrication will dress up the metal-can heaters and prevent rusting.

For a heater larger than 500 ml. an enameled pan makes a good container. However, because of the slanting sides of these pans it is necessary to anchor the cement with at least three nails placed through the sides near the bottom of the pan. Heaters may be designed to fit other vessels such as a beaker, Florence flask, or a funnel.

Figure 3

All of the wire has been covered.

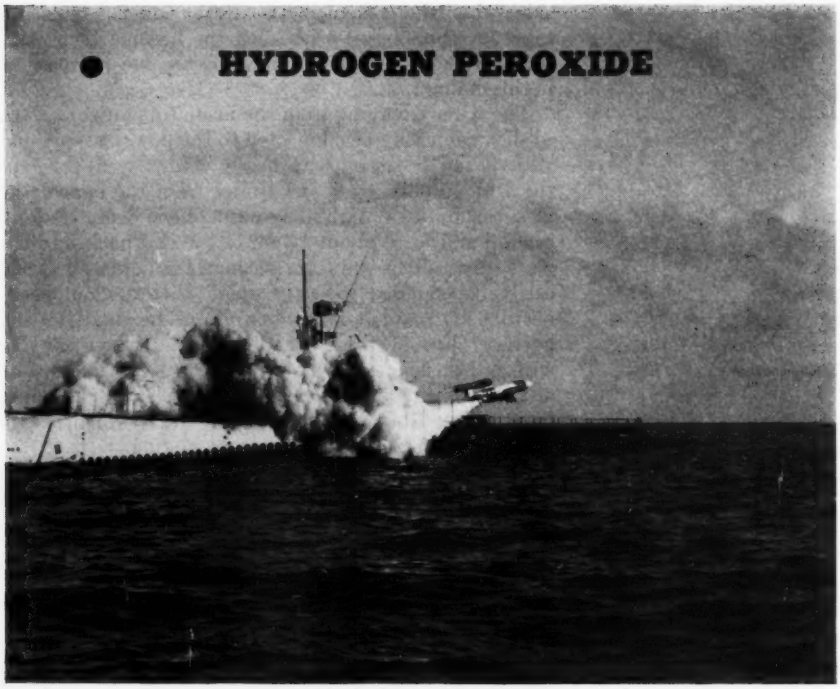
Suitable wire lengths for various sized heaters are:

Flask size, ml.	*B&S*	*Length, ft.*	*Approximate wattage*
50	26	12	450
50	28	12	300
100–300	24	16	600
300–5000	22	22	650

HYDROGEN PEROXIDE

E. S. SHANLEY

Buffalo Electro-Chemical Company, Inc., Buffalo, New York



The Launching of a Navy LTV-N-2 Missile Using Hydrogen Peroxide

In a recent issue of This Journal[1] it was noted that the term "mad as a hatter" was derived from the fact that felt-hat makers used to become quite mad, from chronic mercury poisoning. Not mentioned was the fact that this industrial hazard no longer exists, due to replacement of the mercury with hydrogen peroxide. From hat making to submarine propulsion and from germ killing to raising dough, hydrogen peroxide has found a multitude of uses in modern living. It is the purpose of this paper to describe the manufacture and uses of this versatile chemical.

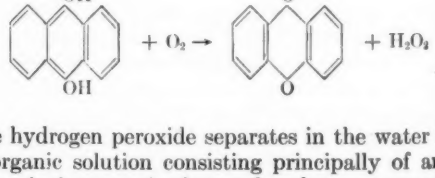
FORMATION AND MANUFACTURE

Hydrogen peroxide is formed in detectable amounts in a great many chemical reactions. The direct combustion of hydrogen in oxygen produces considerable amounts if the flame is quickly cooled. Under certain conditions, hydrogen peroxide can be made the principal product of the direct reaction between hydrogen and oxygen (*1*).

Detectable quantities are formed by cathodic reduction in aqueous solutions containing dissolved oxygen. A "wave" corresponding to this reaction is observed in polarography (*2*). There is a patented process for manufacturing hydrogen peroxide in alkaline solution by cathodic reduction of oxygen gas at a carbon cathode (*3*).

Hydrogen peroxide can be detected in water after ultrasonic irradiation. Presumably the presence of oxygen is also necessary. The silent electric discharge in gas mixtures containing hydrogen and oxygen produces hydrogen peroxide in quantities. The photochemical reaction of hydrogen and oxygen, under appropriate conditions, yields only hydrogen peroxide as the primary product (*4*).

Hydrogen peroxide can be synthesized by cyclic oxidation and reduction of certain easily oxidizable organic compounds such as hydrazobenzene or anthrahydroquinone. When anthrahydroquinone in benzene solution is mixed with water and blown with air or oxygen, the following reaction occurs:



The hydrogen peroxide separates in the water layer. The organic solution consisting principally of anthraquinone in benzene is then reduced to regenerate the hydroquinone. Various reducing means can be used, including hydrogen gas, sodium amalgam, and electrolysis (*5*).

The foregoing methods for hydrogen peroxide synthesis are of no present commercial significance, all production being accounted for by the following two processes.

The barium peroxide method, original commercial procedure for hydrogen peroxide manufacture, involves

[1] J. Chem. Educ., 25, 180 (1948).

the same reactions observed by Thènard in 1818 when he discovered this compound. Barium oxide, roasted in air, is converted in part to barium peroxide. When this compound is dissolved in acid a solution containing H_2O_2 results.

$$BaO + {}^1/_2\,O_2 \rightarrow BaO_2$$
$$BaO_2 + H_2SO_4 \rightarrow BaSO_4 + H_2O_2$$

Sulfuric acid is preferred because it removes the barium from solution, leaving a relatively pure aqueous solution of hydrogen peroxide. At the same time a valuable by-product, *blanc fixe*, is produced. Large quantities of dilute hydrogen peroxide (generally under 10 per cent H_2O_2) are produced commercially by this process (*6*).

The bulk of commercial hydrogen peroxide is produced by the electrolytic oxidation of a bisulfate solution to persulfate.[2] Subsequent hydrolysis and distillation yields a relatively pure and concentrated hydrogen peroxide solution. The reactions can be summarized as follows:

$$2HSO_4^- \rightarrow S_2O_8^{--} + 2H^+ + 2e$$
$$S_2O_8^{--} + 2H_2O \rightarrow 2HSO_4^- + H_2O_2$$

Either sulfuric acid or ammonium bisulfate can be used in this cyclic operation.

The electrolysis is generally carried out in stoneware tanks using platinum anodes. After oxidation of a large part of the bisulfate to persulfate, the solution is fed to stoneware distillation apparatus where the hydrolysis is carried out by heating the solution. Hydrogen peroxide and water are flashed off under vacuum while the spent solution, again consisting of bisulfates, is returned to the electrolysis.

It is customary to take off the hydrogen peroxide from the persulfate process at about 30 per cent H_2O_2. Since hydrogen peroxide boils at a higher temperature than water and since no constant-boiling mixtures are formed, almost any desired concentration can be reached by fractional distillation. The very special problems involved in distilling this material have been successfully solved, as evidenced by the commercial availability of solutions as strong as 90 per cent H_2O_2.

Commercial hydrogen peroxide is shipped in 250-pound aluminum drums, in tank trucks, and in aluminum tank cars of up to 8000 gallon capacity. The glass carboy, formerly the standard container, is disappearing from use. Common commercial strengths are 27.5 per cent, 35 per cent, and 50 per cent by weight H_2O_2, while higher concentrations, including 90 per cent, are today largely limited to special applications. Laboratory reagent grade is sold in glass or polythene bottles usually at 30 per cent by weight H_2O_2.

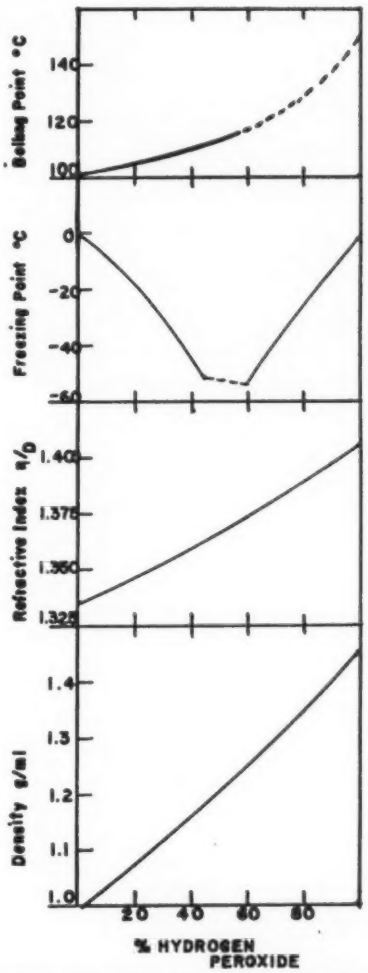
At the point of use hydrogen peroxide is generally stored in the shipping container or in high-purity aluminum tanks. It is often moved through pipe lines by means of aluminum or stainless steel pumps. The lines may be of aluminum, stainless steel, porcelain, or pyrex. It is absolutely necessary to exclude iron and steel, brass, bronze, lead, Monel, and most other metals from the system on account of the decomposing action of these materials on the peroxide.

Certain kinds of plastics are used for gaskets, packings, and flexible connections. Polythene and certain of the polyvinyls and polyfluoroethylene plastics are suitable.

[2] For a description of modern practice see, *e. g.*, ref. 7.

PROPERTIES

A few of the physical properties of hydrogen peroxide solutions are detailed in the figure below.



Some Physical Properties of Aqueous Hydrogen Peroxide

The most characteristic chemical reactions of hydrogen peroxide are those in which it exhibits oxidizing power. Thus, sulfites are oxidized to sulfates; ferrous salts to ferric; titanic salts to pertitanates; alcohols to aldehydes and acids. Many organic dyestuffs and coloring materials are oxidized to colorless compounds. The oxidizing action is relatively mild compared with chlorine, hypochlorites, and permanganate.

Hydrogen peroxide also acts as a reducing agent, for example it reduces manganese from the MnO_4^- state to Mn^{++}.

Another characteristic part of hydrogen peroxide behavior is its tendency to break down into oxygen and water. H_2O_2 is thermodynamically quite unstable, as shown by the free energy and heat of formation.

$$H_2O_{2(g)} \rightarrow H_2O_{(g)} + {}^1/_2\,O_2 \qquad \Delta F = -30 \text{ kg.-cal.}, \quad \Delta H = -23 \text{ kg.-cal.}$$

The decomposition of hydrogen peroxide is an extremely slow reaction in the absence of catalysts. In fact, there is evidence that most of the decomposition is catalytic, occurring only in the presence of foreign material or on the container walls. Very pure solutions in suitable small laboratory containers lose less than 1 per cent of their active oxygen in a year. The actual observed rate is dependent upon the size (surface-volume ratio) of the container, its nature, its previous history, and the purity of the peroxide to a degree hardly measurable by analytical means. In fact, the acceleration of H_2O_2 decomposition by heavy metals is among the most sensitive of analytical procedures.

Most heavy metals and their ions, the enzyme catalase, and all varieties of dust and dirt, catalyze the decomposition of hydrogen peroxide solutions. Because of this sensitivity it is common practice to add stabilizers to hydrogen peroxide solutions. These probably owe their effect to inactivation of decomposition catalysts rather than to any positive effect on the peroxide itself. Typical stabilizers are materials like pyrophosphates or fluorides, able to sequester heavy metal ions, complex-formers like 8-hydroxyquinoline and acetanilide, or adsorbents, for example, freshly precipitated stannic oxide. Sodium pyrophosphate is perhaps the most universally used stabilizer, alone or in combination with other stabilizers. It is customary to use from about 50 to a few hundred parts of $Na_4P_2O_7$ per million parts of peroxide.[3] In dilute "drugstore" peroxide, acetanilide is commonly used. U. S. P. 3 per cent hydrogen peroxide is allowed to contain 400 p.p.m. of acetanilide. Hydrogen peroxide free from additives is available for use as a laboratory reagent. Very concentrated solutions, such as 90 per cent H_2O_2, are made and handled at such extremely high purity that little or no stabilizer is added.

Complementary to, but distinguishable from, the action of catalysts and stabilizers is the action of acids and bases upon hydrogen peroxide. In general, solutions containing acids are less susceptible to catalysis, while alkaline solutions are much more readily decomposed. Hydrogen peroxide itself is a weak acid, dissociating according to the following equation:

$$HOOH \rightarrow H^+ + OOH^- \qquad K_{298} = 2.4 \times 10^{-12}$$

[3] Most technical data on peroxide stabilization are contained in the patent literature. See, *e. g.*, ref. *8*.

The increased concentration of OOH^- ions in solutions containing bases undoubtedly contributes to the instability.

Hydrogen peroxide weaker than about 65 per cent by weight is not especially hazardous, although it is quite corrosive to the skin and eyes. Above 65 per cent the heat liberated upon decomposition is more than enough to evaporate all of the water present and formed. Consequently, steam and oxygen are the products of rapid decomposition. From 90 per cent H_2O_2 the adiabatic decomposition at atmospheric pressure yields for each volume of liquid about 5000 volumes of steam and oxygen at 750°C. H_2O_2 over 65 per cent by weight is capable of igniting combustible materials. Shanley and Greenspan have recently described the hazards and other properties of highly concentrated hydrogen peroxide (*9*).

ANALYSIS OF SOLUTIONS

Hydrogen peroxide solutions are readily analyzed for active oxygen content by means of either their oxidizing or reducing action. Iodide in mildly acid solution is quantitatively oxidized to free iodine by H_2O_2. The reaction is rather slow and it is customary to allow some 10 or 20 minutes for completion. Alternatively, a drop or two of dilute ammonium molybdate solution may be added as a catalyst, whereupon the reaction goes to completion at once. The free iodine is then determined with standard thiosulfate solution in the usual fashion.

The reducing action of hydrogen peroxide on potassium permanganate is very commonly used for the analysis of concentrated peroxide solutions. Permanganate is titrated into the strongly acidified peroxide sample.

$$2KMnO_4 + 3H_2SO_4 + 5H_2O_2 \rightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 5O_2$$

If insufficient acid is used some of the manganese will eventually be reduced only to MnO_2. This material is a vigorous decomposition catalyst for the remaining hydrogen peroxide so the titration is ruined if a brown precipitate appears.

Ceric sulfate can be substituted for potassium permanganate in the above method with entirely analogous results.

Physical methods are also useful for H_2O_2 analysis. The relation between refractive index and composition for the system water-hydrogen peroxide has been fully explored (*10*). Determination of n_D affords an excellent method for estimating concentration, especially useful with strong H_2O_2 solutions in water.

Determination of the density of H_2O_2 solutions can also be used as a means of measuring concentration. This method is handicapped by the tendency of peroxide solutions to release gas bubbles on the surface of floats or containers, with obvious effects on the accuracy of the measurement.

INDUSTRIAL USES

Bleaching of Cotton. (*11*) In 1949 about eight billion yards of cotton cloth were bleached in the United States. Of this vast total, about seven billion yards were bleached with hydrogen peroxide.

In the twenty-five years since its introduction in this country, concentrated hydrogen peroxide has thus nearly displaced the older bleaching agents. Original impetus for this change-over was furnished by the fact that fabrics containing colored portions could be bleached with peroxide without harming the colors. Further development has proved that most cotton fabrics can be advantageously bleached with this reagent.

Cotton may be bleached at any stage in the manufacturing process; in raw stock form, as yarn, or as fabric. The chemical treatment is similar in all cases, the processing machinery being adapted to the form of the material. Tonnage-wise, cloth bleaching is vastly more important than the others.

Cotton bleaching solutions are generally made up to contain from about $^1/_3$ to 1 per cent H_2O_2 and about 1 to 3 per cent sodium silicate (water glass), quantities based upon the weight of cotton. Sodium hydroxide is sometimes added to the formula, especially if the bleaching is to be conducted at low temperatures.

Many different kinds of apparatus are used for bleaching, depending upon the form of the cotton and other factors. Most cotton fabrics are treated by either the cold bleaching process, the kier process, or the continuous process. Fabric for cold bleaching is dampened with a solution containing hydrogen peroxide, sodium silicate, and sodium hydroxide, then rolled up in the open width to stand for some hours at or near room temperature.

In kier bleaching the fabric is packed into kiers (large iron kettles) holding a few tons of cloth. The treating solution, containing hydrogen peroxide and sodium silicate, is circulated and warmed by external pumps and heaters. Bleaching is complete in some six to fifteen hours, often at around 180°F. Two or more such treatments, or one "boil" with sodium hydroxide followed by a peroxide and silicate treatment, may be required to obtain a full white.

The continuous process, most modern method for bleaching cotton fabrics, has revolutionized this industry during the ten years since its introduction. In this method the cloth is passed in a continuous run through a saturator and dampened with approximately its own weight of a bleaching solution containing about 0.3 per cent hydrogen peroxide and about 1 per cent sodium silicate. The dampened cloth is piled continuously into the top of a chute shaped like the letter J and heated to 212°F. with steam. The packed cloth requires about one hour to travel through the chute to the lower opening, where it is pulled off continuously through a washing machine. Such a unit can produce bleached cloth continuously at a rate limited only by the washing machine. Speeds ranging from 80 to 360 yards per minute are commonly used, corresponding to production rates as high as 8000 pounds of bleached cloth per hour. Various pretreatments and after-treatments are usually connected in series with the bleaching unit. The new system has the usual advantages of continuous processing as contrasted with batch, such as greater uniformity, lower processing costs, simpler routing of material, and lower inventory of goods in process. Bleaching with this method can be carried out for a total chemical cost around two to three dollars per thousand pounds of cloth. The trade journals have carried extensive reviews of this new process (*12*) which is a far cry indeed from traditional bleaching methods of the past (*13*).

Bleaching of Wool. Very large quantities of wool are bleached with peroxide during the manufacture of finished articles. Raw wool for carpet yarns and other purposes is often bleached concurrently with the scouring process. The stock received by the woolen mill, nearly half foreign matter, is cleaned in a continuous scouring operation using detergents and soda ash (sodium carbonate). Hydrogen peroxide is often added to the last rinse water in this operation just before the wool stock is squeezed and dried. Bleaching occurs largely during drying. No further treatment is necessary since the peroxide leaves no residue on the wool. Such bleached wool is generally used for dyeing the lighter colors where it gives brighter, cleaner shades.

Woolen piece goods, knitting yarns, knitted fabrics, and the like, are often bleached by several hours' immersion at 120°F. in a bath containing about 1 per cent H_2O_2, and a small amount of sodium pyrophosphate and ammonia. This so-called standing bath is often made up to strength and used over and over again. A great deal of wool is also bleached by simply dampening with alkaline hydrogen peroxide and then piling up for several hours' storage at room temperature. A washing step completes the process.

Bleaching of Pulp and Paper. Recent developments in the bleaching of groundwood pulp have revolutionized this industry. Groundwood pulping, *i. e.*, mechanical disintegration of whole logs, produces about twice as much paper from a cord of wood as does chemical pulping. In addition, groundwood operations cause almost no stream pollution, in contrast to the extremely heavy pollution from conventional sulfite pulping operations. However, groundwood is quite dark in color and cannot be bleached by the older pulp bleaching methods. Successful methods for bleaching this pulp with peroxides have recently been worked out, so that the product is light enough in color for inclusion in magazine and book papers. Both hydrogen peroxide and its near relative sodium peroxide are now being used on a large scale for this purpose. During 1950 some 300,000 tons of groundwood and similar pulps were peroxide bleached and supplanted a like quantity of chemical pulp. To make this same weight of chemical pulp would require about twice

as much wood, so that the new bleaching process is to be credited with saving the equivalent of a small forest in 1950 alone. The groundwood bleaching process involves mixing a few tenths of a per cent of hydrogen peroxide or an equivalent amount of sodium peroxide and about 5 per cent sodium silicate (42° Bé) with the slurry of pulp and water. Depending upon the equipment used, the pulp slurry may consist of 97 parts water to 3 parts pulp (so-called low density system) or as little as 80 parts water to 20 parts of pulp. In this last case the mixture resembles wet blotting paper. Bleaching is complete in one to six hours at about 150°F. High-density bleaching gives the most bleach for the chemicals but requires costly equipment.

Peroxide bleaching is also being used on an expanding scale for the bleaching of chemical pulps where it supplants or supplements chlorine bleaching.

Bleaching of Other Materials. Hydrogen peroxide is perhaps the most universally useful decolorizing and bleaching agent. In addition to the materials described above, peroxide is used in bleaching such diverse materials as straw, leather, feathers, gelatin, glue, buttons, waxes and oils, fats, soap, and many others. Its use in bleaching human hair is known to all. Blond furniture is equally indebted to hydrogen peroxide, the commercial method for bleaching woods consisting of a treatment with hydrogen peroxide and an alkali, usually brushed or sprayed on the surface. The hydrogen peroxide is sometimes used at full 35 per cent strength on the wood surface. The alkali may be mixed with the peroxide solution or applied separately. It usually contains sodium silicate and often sodium hydroxide as well. In this process the wood is bleached in a thin surface layer only. Wood veneers are sometimes bleached by immersion in a more dilute alkaline peroxide solution, in which case the bleaching action penetrates through the sheets.

Carroting of Fur. The making of felt, one of the oldest of man's arts, is carried out by subjecting animal hair to mechanical action in the presence of moisture, thus causing the individual fibers to interlock and adhere. Some animal fibers have considerable natural felting ability, but in general chemical treatment of the hair is required to enhance this property before good felt can be made. Solutions of mercuric nitrate in nitric acid came to be universally used for this purpose. The name "carroting" was applied because of the orange-yellow color imparted to light-colored fur. Workmen employed as fur carroters often became victims of chronic mercury poisoning, involving loss of teeth, excessive salivation, and progressive loss of muscular control. In former times, it is said, workmen were actually carried to their benches, so badly crippled by the "shakes" as to be incapable of walking. The "mad as a hatter" simile was founded upon this lack of muscular coordination and not upon actual mental derangement.

Hydrogen peroxide was first used in carroting in an effort to improve the color of light furs which had been yellowed by the mercury carrot. Then it was discovered that hydrogen peroxide had carroting properties of its own. Extensive development led to a successful nonmercuric carrot, containing nitric acid and hydrogen peroxide as the active ingredients (*14*). This development was quickly followed by legislation outlawing the mercuric carrot, so that the Hatter's Shakes and the Mad Hatter are gone forever, welcome casualties to technical progress.

Manufacture of Other Peroxides. Hydrogen peroxide may be looked upon as the parent compound of a whole series of organic peroxides. These are made by substituting organic radicals for one or both of the hydrogen atoms. If one hydrogen is replaced by an alkyl group, a hydroperoxide, the "per" analogue of an alcohol, is produced. *t*-Butyl hydroperoxide $(CH_3)_3C$-OOH, for example, is an article of commerce. Diperoxides, the "per" analogues of ethers, are also known. These are probably the explosive compounds which form spontaneously in ethyl ether, isopropyl ether, etc., especially upon exposure to air and sunlight.

Acyl groups may also be substituted for the hydrogen atoms in peroxide. Substitution of one hydrogen atom in favor of an acetyl radical gives peracetic acid, CH_3CO-OOH. This is one of the most useful of organic peroxides, employed in food and equipment sanitizing and mold inhibition as well as in many special bleaching applications. Peracids are usually formed by reaction of the organic acid with very concentrated hydrogen peroxide.

Diacyl peroxides are formed by reacting the corresponding acid anhydride or chloride with alkaline hydrogen peroxide. Dibenzoyl peroxide, $(C_6H_5CO_2)_2$, the most widely used organic peroxide, is used in flour bleaching and in polymerization reactions. Diacetyl peroxide, extremely explosive in the pure state, can be tamed by making and handling in solution. In this form it finds use as a polymerization promoter.

Inorganic peroxides such as calcium, strontium, magnesium, and zinc peroxides can be made by reacting soluble salts of these metals with aqueous alkaline hydrogen peroxide. Under these conditions the corresponding peroxides, CaO_2, MgO_2, etc., precipitate in good yield and purity. These compounds resemble the corresponding oxides in appearance and in physical properties. They are quite stable in storage, insoluble in water but soluble in acid to give solutions containing the soluble metal salt and hydrogen peroxide. These compounds are used in commercial baking, in pharmacology, in dentifrices, in tracer bullets, and in a variety of other ways.

A number of inorganic perhydrates are also made from hydrogen peroxide. Typical examples with approximate compositions are given below:

Sodium carbonate perhydrate	$2Na_2CO_3.3H_2O_2$
Sodium pyrophosphate perhydrate	$Na_4P_2O_7.2H_2O_2$
Urea peroxide	$CO(NH)_2.H_2O_2$

Urea peroxide is used as a convenient solid source of

hydrogen peroxide in hair dyeing, etc., the others find use in dental plate cleaners, scouring preparations, etc.

Chemical Synthesis. Hydrogen peroxide by itself or converted to a peracid is a valuable reagent in organic synthesis. A few typical reactions are listed below. Many additional reactions are described in the literature (*15*).

Hydroxylation of double bonds

$C_8H_{17}CH{:}CHC_7H_{14}COOH \xrightarrow{H_2O_2,\ \text{Acetic acid}} C_8H_{17}CH(OH)CH(OH)C_7H_{14}COOH$

Oleic acid — Dihydroxystearic acid

Preparation of disulfides

$C_2H_5SH \xrightarrow{H_2O_2} C_2H_5SSC_2H_5$

Ethyl mercaptan — Diethyl disulfide

Preparation of Quinones

$C_{10}H_7CH_3 \xrightarrow{H_2O_2} C_{10}H_7CH_3O_2$

2-Methyl naphthalene — 2-Methyl 1,4-naphthoquinone

Halogenation

$O{:}C_6H_4{:}O \xrightarrow[HCl]{H_2O_2} O{:}C_6Cl_4{:}O$

Benzoquinone — Chloranil

Epoxidation

$C_8H_{17}CH{:}CHC_7H_{14}COOH \xrightarrow[\text{Acetic acid}]{H_2O_2} C_8H_{17}\underset{\diagdown\,O\,\diagup}{CH{-}CH}C_7H_{14}COOH$

Oleic acid — Epoxystearic acid

Production of Porous Articles. Since hydrogen peroxide is readily broken down into oxygen and water and since the time and rate of breakdown can be controlled, this chemical is often used as a leavening agent. In food products the oxidizing action is generally undesirable, but this is not a drawback in the case of other materials. Large quantities of porous gypsum boards have been made in this way. The expanded material is desired because of its light weight and its sound-deadening and heat-insulating properties. Some quantities of foam rubber are also made with the aid of hydrogen peroxide. The technique usually consists in combining the peroxide with the material to be foamed in a mildly alkaline medium. A decomposition catalyst is added when the foaming action is desired. Heavy metal salts (lead, cobalt, manganese) or catalase-containing solutions are often used.

Production of Power. In World War II hydrogen peroxide was used in connection with a number of devices for power generation. Some German applications which have been publicized include the following:

The Buzz bomb, or V-1, was launched from a catapult-like device. Hydrogen peroxide at about 80 per cent concentration was injected into a cylinder together with a strong permanganate solution. Steam and oxygen from the resulting decomposition activated a piston to which the V-1 was attached.

The German ME 163, a rocket-propelled, manned aircraft was powered solely by decomposing 85 per cent H_2O_2. Later models used a fuel with the hydrogen peroxide so as to obtain more power. These planes, while of very limited range, were spectacular performers, said to be capable of climbing 30,000 feet per minute.

In the V-2 long-range rocket, an unmanned missile, decomposing 90 per cent hydrogen peroxide was used as a source of steam to operate the fuel pumps. These pumps moved nearly five tons of alcohol and liquid oxygen from the supply tanks to the rocket jets during the first minute or so of flight. After consumption of all the fuel, the missile continued in free flight at a maximum velocity of about 3500 miles per hour.

Experimental submarines were operated at speeds up to 30 miles per hour submerged using the so-called Walther cycle engine. Heat was obtained by burning fuel oil in the atmosphere provided by decomposing 80 per cent hydrogen peroxide. The resulting hot gases, principally steam and carbon dioxide, were used to operate a turbine from which the motive power was taken. Available submerged shaft horsepower and shaft horsepower hours were vastly greater than for the conventional battery-operated sub. The peroxide was carried in huge plastic bags outside the pressure hull.

Laboratory Reagent. A substantial quantity of high-purity hydrogen peroxide is used as a laboratory reagent. Some inorganic analytical procedures which depend upon this reagent include colorimetric methods for titanium and vanadium and various steps in analyses for iron, chromium, tungsten, molybdenum, and others. A standard method for continuously recording sulfur dioxide concentration in air involves absorption of the SO_2 in dilute hydrogen peroxide followed by measurement of the electrical conductance of the resulting sulfuric acid solution.

In the organic laboratory hydrogen peroxide is used in Kjeldahl digestions, in determination of sulfur in certain organic compounds, in measurement of catalase activity of blood, body fluids, and the like, and in determination of formaldehyde and certain phenols to name a few typical cases.

Other Uses. The special properties of hydrogen peroxide are used in surprisingly diversified ways. A few illustrative examples are listed below.

Substantial quantities of hydrogen peroxide are still used in disinfecting and cleansing of wounds, in mouth washes, and for other similar uses. There is renewed interest in hydrogen peroxide as a general-purpose antiseptic. Hydrogen peroxide is a very rapid and efficient antichlor and has been used for this purpose in swimming pools, in textile and sugar processing, etc., to destroy residual active chlorine. In the same way, traces of sulfur dioxide can be removed from various materials. Hydrogen peroxide is used to oxidize and thereby fix on the fiber the so-called vat or ingrain dyestuffs. The oxidizing action of peroxide is also put to use in blueprint developing solutions and in the ripening of alkali cellulose for viscose manufacture. Another special use is in the purification of metal salt solutions—for example, nickel plating solutions—

where peroxide additions are used to oxidize the iron content to the ferric state. The ferric iron, insoluble at the existing pH, is filtered off as ferric hydroxide.

SUMMARY

Hydrogen peroxide technology furnishes a prime example of the beneficial effects which stem from industrial research. Twenty-five years ago 27.5 per cent hydrogen peroxide was something of a rarity. Since then ways have been devised to make the material purer, more stable, more reliable in action, and reasonable in price. The old green glass balloon carboy, transported in a basket of hay, has given place to the modern aluminum tank car and tank truck. In addition to improving the product and its containers, hydrogen peroxide manufacturers have gone to remarkable lengths in devising new processes and improving old ones where their product might find use. Nearly every major use of this chemical has been pioneered by the manufacturers seeking to enlarge their markets. Some of the fruits of this policy are the continuous and other bleaching processes, the groundwood pulp bleaching process, and the techniques and reagents now available for organic epoxidations. These things represent gains to the peroxide manufacturer, to industries employing hydrogen peroxide, and to the general public, consumers of the ultimate products.

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A RAPID TEST FOR CASTOR OIL IN SOAPS AND LIPSTICKS[1]

SISTER M. CLARITA MANGOLD
Rosary Hill College, Buffalo, New York

Vizern and Guillot[2] present a method for the characterization of castor oil in mixtures of fatty materials. It is based on the fact that ricinoleic acid (the chief fatty acid of castor oil) with excess of alkali forms capryl alcohol (*sec.* octyl alcohol), one of the eighty-nine isomers of octyl alcohol.

$$\underset{\text{Ricinoleic Acid}}{CH_3(CH_2)_5CHOHCH_2CH{=}CH(CH_2)_7COOH} \xrightarrow{\text{excess NaOH}} \underset{\text{Sodium Sebacate}}{NaO_2C(CH_2)_8CO_2Na} + \underset{\text{Octyl Alcohol}}{C_8H_{17}CHOHCH_3}$$

Octyl alcohol has a peculiar, strong, aromatic odor which cannot easily be mistaken. This method was tried on castor oil "as such," and on lipsticks with known amounts of castor oil and found to be valid.

However, the author discovered that the method may be greatly simplified and the test applied in a few minutes with very small amounts of materials. There is no need of separating castor oil from other constituents. It is not necessary to carry out the long process of saponification nor to follow the procedure given by Vizern and Guillot. It was found that other oils, fats, waxes, dyes, fillers, perfumes, etc., did not interfere with the formation and identification of octyl alcohol.

PROCEDURE

Put about one gram of a soap or lipstick into a test tube, add three pellets of sodium or potassium hydroxide and heat gently, shaking occasionally until heavy white fumes form. Cover, and let cool, then smell the contents. (The odor of octyl alcohol can be detected even when hot.) The odor of octyl alcohol indicates the presence of castor oil. If water is added to the test tube when cool, the odor is still more pronounced.

This rapid test was applied to the pure oil, to laboratory lipsticks and soaps having varying amounts of castor oil, and to six commercial lipsticks. All these, except one commercial lipstick, responded to the test. The laboratory lipsticks and soaps were purposely highly perfumed to find out if this interfered with the odor of octyl alcohol. As low as 10 per cent of castor oil was detected by this method.

[1] Partly taken from a thesis submitted to St. Bonaventure College, St. Bonaventure, New York (1949).
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CHEMISTRY AND THE SPECTRUM BEFORE BUNSEN AND KIRCHHOFF[1]

TILLMON H. PEARSON and AARON J. IHDE
University of Wisconsin, Madison, Wisconsin

PRODUCTION of colors by refraction was known to Seneca (*1*). Kepler (*2*) made use of an equilateral glass prism for the study of the subject. The Archbishop of Spalatro, Marco Antonio de Dominis (*3*), explained the rainbow by postulating that the light reflected from the inner surfaces of raindrops was colored by passing through different thicknesses of water. Descartes (*4*) improved the explanation and made it in terms of refrangibility. He also calculated the angle of the bow.

It was Newton (*5*), however, who recognized the significance of the phenomenon. He wrote: "I procured me a Triangular glass-Prisme to try therewith the celebrated *Phoenemena of Colours.*" After sunlight from a circular hole in the shutter had passed through his prism he observed, as others had before him, an elongated and rainbow-hued spot on the wall. His search for understanding was aided by a second prism which revealed that the various colors of the spectrum could be refracted again but without further change in color. The colors of the blue end were refracted most and those of the red end least, just as had happened when the light passed through the first prism. Newton also observed that the second prism could be used to recombine into white light the colors separated by the first prism.

The ideas of Newton on light were immediately attacked. Robert Hooke, who had a color theory of his own as the result of his work on thin films, was especially vociferous in his denunciation. The reception of his first scientific paper caused Newton to withdraw from scientific discussion. Most of his later publications came only after prolonged persuasion on the part of his friends. It is of interest to note that his book, "Optiks," was only published in 1704, the year after Hooke's death.

The eighteenth century saw little progress in the further understanding of light. Scheele (*6*) in 1777 reported the effects of spectral colors on the darkening of silver chloride. It was well known among silver miners that a certain ore found as a white mineral, horn silver, turned dark upon exposure to sunlight. Scheele exposed samples of the chloride to the various colors of the spectrum, noting that the darkening was most rapid at the violet end. His results were verified and extended in 1801 by Johann Wilhelm Ritter (*7*) who found the dark region beyond the violet portion of the spectrum still more effective in producing darkening than the visible violet. The same discovery was made independently by William Hyde Wollaston (*8*).

It was the discovery of the infrared region of the spectrum a year earlier which had prompted Ritter to investigate the possible existence of radiant energy beyond the violet. The famed astronomer, William Herschel (*9*) in studying the illuminating and heating power of the various colors of the spectrum observed a steady increase in heating power from the violet to the red. He used the arrangement illustrated (Figure 1). The blackened bulb of thermometer 1 was placed in the color range under test while thermometers 2 and 3 to the side of the spectral area served as controls. When the bulb of thermometer 1 was placed in the dark region beyond the visible red it showed an even faster temperature rise than it did when the red color fell upon it, revealing the passage of radiant heat of low refrangibility through the prism.

Wollaston's paper (*8*) verifying the existence of ultraviolet light also reported the presence of dark lines in the spectrum of sunlight. Up to this time investigators had been working with highly impure spectra resulting from circular openings or wide slits admitting the beam of light. Wollaston used a narrow slit, not more than a twentieth of an inch in width. When he viewed the light from the slit at the distance of 10 or 12 feet, through a flint-glass prism held near the eye, he saw the beam separated into red, yellowish

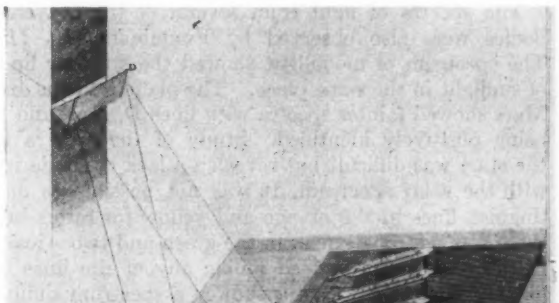

Figure 1. Herschel's Recognition of Infrared

[1] Presented before the Division of the History of Chemistry at the 118th Meeting of the American Chemical Society, Chicago, Illinois, September, 1950.

green, blue, and violet. In the resulting spectrum he reported seven dark lines. Five of the lines were reported as being on the boundaries of the colors observed, but the other two were observed within the yellowish green and the blue regions.

These lines, plus many additional ones, were independently rediscovered in 1814 by Fraunhofer (*10*). The young lens manufacturer, in studying the refractive index of glass samples for particular colors, noticed a bright double line (of sodium) in the spectrum of the flame which he was using. This same double line was observed in alcohol and sulfur flames as well as in oil and tallow light in exactly the same position and was therefore useful in the determination of indexes. In order to find out if a similar line could be observed in the solar spectrum, he allowed sunlight to pass through a narrow slit and fall upon a flint-glass prism set in front of a telescope. Instead of a bright yellow double line he observed countless vertical lines darker than the colored parts of the spectrum in which they occurred. Eight of the most prominent lines were labeled *A* to *H*, and nearly 600 lines were observed, all but the less definite ones being mapped (Figure 2).

Figure 2. Fraunhofer's Map of the Solar Spectrum

He reported on the effect of slit width and types of prisms on spectral purity. His experiments convinced him that the lines were due to the nature of sunlight rather than to diffraction caused by the slit or to optical illusions. During the remaining years of his life he continued his studies of spectra without ever realizing the significance of the lines which today bear his name.

The spectra of light from several of the heavenly bodies were also observed by Fraunhofer (*10*, *11*). The spectrum of moonlight showed the stronger lines of sunlight in the same place. The planets Venus and Mars showed fainter spectra with lines *D*, *E*, *b*, and *F* being positively identified. Study of the spectra of the stars was difficult but revealed a lack of similarity with the solar spectrum. It was not possible to distinguish lines in the orange and yellow for Sirius but there was a strong streak in the green and two streaks in the blue which seemed unlike any of the lines in planetary spectra. Castor showed a spectrum similar to Sirius. The *D* line was identified in the spectrum of Pollux and Betelgeuse and possibly in that of Procyon.

In 1821 Fraunhofer (*12*) reported experiments with diffraction gratings. His first gratings were made of wires wrapped closely around a flat frame. Later he made gratings by ruling lines on glass covered with gold foil. With his gratings he determined the wave length of the line labeled *D*. With gratings made of wires ranging between 0.04 and 0.6 mm. in thickness he obtained values lying between 0.0005882 and 0.0005897 with a mean of 0.0005888 mm. The accepted modern value is 0.0005896 mm. for D_1 and 0.0005889 mm. for D_2 (*13*).

The phenomenon of diffraction, first reported in 1665 in the posthumous treatise of the Jesuit mathematics professor at Bologna, Francesco Grimaldi (*14*), had received little attention during the century in which Newton's corpuscular theory of light had been dominant. Now, with the re-emergence of the undulatory theory as a result of the work of Young and Fresnel, diffraction took on a new significance and the diffraction grating was destined to play an important role in the study of spectra.

The extension of Fraunhofer lines into the infrared was shown by John Herschel (*15*), son of William, in 1840. He covered a paper with gum and lampblack to make it readily absorptive of heat. After dipping in alcohol the paper was exposed to the spectrum of the sun. The alcohol, instead of evaporating evenly in the dark region beyond the visible red, as would have been the case if the spectrum were continuous, evaporated unevenly, leaving several moist patches which indicated that the dark lines apparently carried beyond the visible spectrum. Later experiments with more refined techniques soon verified this assumption.

David Brewster (*16*) reported in 1834 on lines produced by sunlight passed through "nitrous acid gas." He stated as the object of his inquiries, "the discovery of a general principle of chemical analysis in which simple and compound bodies might be characterized by their action on definite parts of the spectrum." After first observing that sulfur attacked the violet end of the spectrum of lamp light and iodine vapor the middle part, he turned his attention to the brown oxide of nitrogen. The spectrum was crossed by hundreds of lines or bands. Upon increasing the thickness of the gas the lines grew more distinct in the red and yellow region and broadened in the blue and violet. The same effect was noted when the thickness of the gas remained constant but the gas was heated, an experiment with attendant dangers since the tubes frequently exploded. Brewster reported the noncoincidence of the lines with the lines of the solar spectrum and the absence of the lines in Fraunhofer's map.

Brewster's work was immediately extended by John Frederic Daniell and William Hallows Miller (*17*) who passed the light of a gas lamp through halogen vapors before examining the prismatic spectrum with a small telescope. In the spectrum of bromine vapor in air they observed the colors interrupted by more than 100 lines. When the concentration of bromine was increased, the blue end disappeared while the lines in the red grew stronger. They also investigated the absorption of iodine vapor and chlorine.

The investigation of absorption spectra was resumed some years later by William Allen Miller (*18*). None of the fourteen colorless gases studied showed lines. When colorless elements were united, however, they

might form colored gases which did show lines while colored elements like iodine might show no lines when in gaseous compounds.

The characteristic bright lines in the spectra of certain metallic salts on heating were first observed by Thomas Melvill (*19*) in 1752. This young Scottish investigator died during the next year and his observations apparently went unnoticed. The difference in flame colors given by sodium and potassium salts appears to have been used as a means of distinguishing salts of these elements by Marggraf (*20*) by 1758. John Herschel (*21*) reported in 1822 that the flame colors of the chlorides of strontium, calcium, barium, and copper, and that of boric acid were resolved on passing through a prism, bright lines being apparent on a dark background. He believed the bright lines would be useful in detecting small quantities of a substance and touched upon the subject in his "Treatise on Light."

Henry Fox Talbot (*22*) suggested in 1833 the use of common salt in the flame of a spirit lamp to obtain homogeneous light. If a stream of oxygen were directed on the salt through a blowpipe a light of great intensity might be obtained. "Strontian and barytes" were also suggested as sources of homogeneous light. During the next year this same investigator (*23*) pointed out that lithium and strontium, both giving red flames, could be distinguished by passing their light through a prism. The strontium flame revealed many red lines plus an orange and a blue line whereas the lithium gave only a single red line. In a subsequent paper (*24*) Talbot referred to the fixed character of the lines of sodium and reported that the spectrum of copper was full of dark lines. Here he was mistaking bright lines for dark lines. He also observed lines in the spectrum of the boric acid flame.

In 1835 Charles Wheatstone (*25*) published the results of his experiments on the spectra of electric arcs produced by metallic electrodes. He noted that bright lines were produced which were characteristic of the metal making up the electrode, observations being made on mercury, zinc, cadmium, tin, bismuth, and lead.

The developments of Niepce, Daguerre, and Talbot, which were leading to photography, naturally found their way into studies of spectra. In 1842 A. Edmond Becquerel (*26*) photographed the solar spectrum with nearly all of the lines registered by the eye of Fraunhofer plus extension into the ultraviolet. John William Draper (*27*) photographed the spectrum only a few months after Becquerel. Draper, occupant of the chair of chemistry and physiology at New York University, had introduced the daguerreotype into portraiture in the United States (*28*). In 1843 he photographed the diffraction spectrum using a grating ruled on glass by John Saxton, a mechanician at the U. S. Mint.

In 1847 Draper (*29*) published results of studies on hot bodies, reporting that all solids become incandescent at the same temperature. Below 525°C. invisible rays were emitted. Above that temperature rays of progressively greater refrangibility were emitted as the temperature increased. All spectra of incandescent solids were continuous.

During all of this period there was no real recognition of the relationship between bright-line and dark-line spectra. Fraunhofer had observed that the bright lines of the sodium flame corresponded to the position of the *D* line in the solar spectrum. Investigators were encountering confusion, however, on at least two scores. On the one hand they were not obtaining bright-line spectra free of the continuous spectrum. Their flames contained sufficient organic material to provide incandescent carbon giving white light. Draper (*30*), in his study of flame spectra, reported that all flames gave a continuous spectrum. On the other hand their flames and even their electric arcs were contaminated with sodium so that the *D* line was present regardless of the element under study.

In spite of this, the decade preceding Bunsen and Kirchhoff's successful work saw a number of investigators groping in the right direction. Foucault (*31*) examining the spectrum of an arc between charcoal electrodes, noticed a line similar to the *D* line of the solar spectrum. Lacking suitable instruments for independent measurement of the lines, he attempted to superimpose the two spectra by passing the sun's rays through the arc and through the prism. This proved that the lines were indeed identical in position. Further, the *D* line of the sunlight was strengthened by passage through the arc. If the sunlight was shut off the *D* line was in the arc source as a bright line. Foucault wrote, ". . . the line *D* appears darker than usual in the solar light . . . Thus the arc presents us with a medium which emits the *D* rays on its own account, and which at the same time absorbs them when they come from another quarter." Foucault failed to follow up his observations on spectra when he devoted his energies to other researches.

In America, David Alter (*32*) was investigating the spectra of numerous metals and gases by 1855. In describing the spectra of hydrogen and other gases he stated that each element had a characteristic spectrum and that "the prism may also detect the elements in shooting stars." William Swan (*33*), in 1857, called attention to the extremely minute quantities of sodium necessary to bring out the *D* line.

In spite of the findings which anticipated the report of the Heidelberg scientists in 1859, it was the collaboration of Bunsen, the chemist, and Kirchhoff, the physicist, which attracted attention to the significance of the spectrum. Collaboration of these two workers representing different training and objectives, paved the way for a new impetus in chemistry, in physics, and in astronomy.

Bunsen had been making use, in qualitative analysis, of the flame colors imparted by different metallic salts to the colorless Bunsen burner flame. In order to distinguish between similar flames he had viewed them through colored solutions or glasses. One of his

English students, R. Cartmell (*34*), acknowledged Bunsen's valuable suggestions in a paper on the subject. Kirchhoff, at about this time, pointed out that one might better pass the light through a glass prism and view the spectrum. In acting upon the suggestion the pair (*35*) developed their spectroscope for viewing flame colors. The instrument represented no new principles or devices which had not been in use for years, but brought together on one stand the necessary collimating and viewing telescopes with the prism enclosed inside a blackened box (Figure 3).

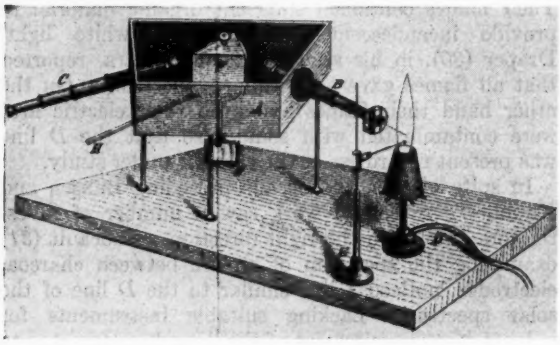

Figure 3. Bunsen and Kirchhoff's Spectroscope

Kirchhoff had already turned his attention to the solar spectrum and published a paper (*36*) on the relation of bright lines to the Fraunhofer lines. When common salt was heated in a flame the characteristic bright line was observed but when white light from incandescent lime, which was known to show a continuous spectrum, was passed through the sodium flame and then through the prism a dark line appeared in the *D* position. Kirchhoff grasped the fact that hot gases absorbed the same kind of light which they emitted. Sodium vapor must be present in the outer atmosphere of the sun, absorbing the *D* line present in the white light coming from the incandescent surface of the sun. When the sun's light was passed through a lithium flame before entering the spectroscope, a new dark line appeared between the Fraunhofer *B* and *C* positions which could not be identified with the known lines of Fraunhofer. Hence, no lithium was present in the sun's atmosphere. Kirchhoff reserved to himself and Bunsen the right to further investigate these phenomena. The first paper was soon followed (*37*) by a long mathematical treatment of the relation between the radiating and absorbing powers of substances for heat and light.

The spectroscope, together with the Kirchhoff laws of spectroscopy, thus provided a powerful new tool for the study of the chemical composition of solar and stellar atmospheres. Astronomers quickly brought the instrument into widespread use. At the same time the earthly uses of the instrument were not overlooked. Bunsen and Kirchhoff (*38*) quickly reported the discovery of the alkali metals cesium and rubidium. William Crookes (*39*) reported evidence for thallium at about the same time. In subsequent years the spectroscope loomed large in the discovery of new elements as well as in more prosaic qualitative analysis.

Immediately after the report of Kirchhoff in late 1859 it was pointed out by E. Verdet (*40*) and by George Gabriel Stokes (*41*) that Foucault had anticipated his discovery. Kirchhoff (*42*) denied prior knowledge of the work of Foucault. William Thompson (Kelvin) (*42*), in correspondence with Kirchhoff, pointed out that Stokes had been offering essentially Kirchhoff's explanation to his students at Cambridge for a number of years but had never bothered to publicize his ideas. Stokes (*43*) later verified this but insisted that, "I have never attempted to claim for myself any part of Kirchhoff's admirable discovery, and cannot help thinking that some of my friends have been overzealous in my cause." John Tyndall (*44*) reiterating the fact that Foucault, Stokes, and Thompson had been close to the discovery, also considered himself on the right track in 1859 if his experiments had not been interrupted, and considered Ångström (*45*) closest to having made the discovery. Crookes (*46*) put forth claims of priority for W. H. Miller.

Kirchhoff (*47*) persistently defended the originality of his own work and attempted to show that the earlier investigators were guilty of misinterpretation of results or confusion of objectives. Nevertheless, in reviewing the growth of understanding of chemistry and the spectrum, it again (*48*) becomes evident that discovery is a cooperative enterprise. Bunsen and Kirchhoff represent the culmination of a long series of steps toward understanding. As a pair they were able to draw together the threads of tradition in order to make the final synthesis. Together they were able to carry their synthesis into new and fruitful investigations in the fields of chemistry and astronomy. Earlier workers had not seen the picture in its broader aspects, had not received suitable publicity for their results, and had not carried on to the point of significant application.

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NATIONAL COLLEGE TESTING PROGRAM

The Committee on Examination and Tests of the Division of Chemical Education of the A. C. S., under the chairmanship of Theodore A. Ashford, announces the 1951 testing program. This year's program differs somewhat from preceding years in that the test user is not invited to one form of the test in a given field, but may choose among all the available forms of the various tests. The following tests are available.

General Chemistry
Form 1948, five parts, 110 minutes.
Form Z, six parts, 113 minutes.

Qualitative Analysis
Form Y, three parts, 100 minutes.
Form Z, three parts, 100 minutes.

Quantitative Analysis
Form X, two parts, 100 minutes.
Form Y, two parts, 110 minutes.

Organic Chemistry
Form W (Revised 1946), two parts, 100 minutes.
Form Y, two parts, 100 minutes.

Physical Chemistry
Form W, five parts, 100 minutes.

Biochemistry
Form X, two parts, 100 minutes.
Form Z, two parts, 100 minutes.

For each of these tests national norms have been obtained and will be mailed to test users with the tests. Consequently the user can interpret the results immediately and need not send the answer sheets to the Educational Testing Service for scoring.

Three tests are prepared by the cooperative effort of over 300 professors who are actively engaged in teaching. The construction of the tests are under the direction of the Committee on Examination and Tests in cooperation with the Educational Testing Service.

According to an agreement reached at the Cleveland meeting between the Committee and the E. T. S., the tests are no longer to be distributed by the latter from Princeton. They, or any further information concerning them, may now be obtained from the Committee Chairman, Theodore A. Ashford, Department of Chemistry, St. Louis University, 1402 South Grand Boulevard, St. Louis 4, Missouri.

AN AUTOMATIC MERCURY VACUUM STILL

W. A. CANNON
Wyandotte Chemicals Corporation, Wyandotte, Michigan

In connection with the purification of mercury for polarographic use, a small mercury vacuum still of conventional design had been used for final vacuum distillation. The utility of this unit was greatly increased by converting it to automatic operation so that it can operate over extended periods of time without periodic attention.

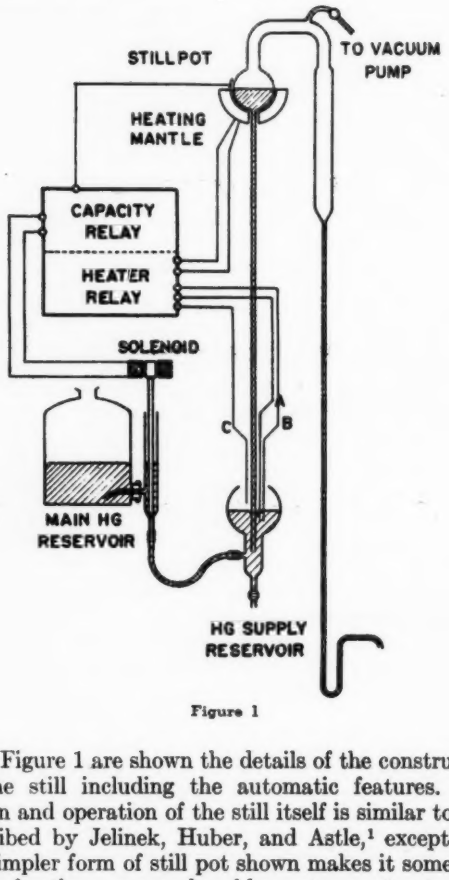


Figure 1

In Figure 1 are shown the details of the construction of the still including the automatic features. The design and operation of the still itself is similar to that described by Jelinek, Huber, and Astle,[1] except that the simpler form of still pot shown makes it somewhat easier for the average glass blower to construct. The still pot is made from a 125-ml. round bottomed flask and is connected to an air condenser 25 mm. in diameter and 30 cm. long. A commercial laboratory heating mantle supplies heat for the distillation. A small hole must be made in the bottom of the mantle to accommodate the supply tube. Care should be taken that the resistance wires are not damaged in the process of making the hole in the heating mantle. The supply reservoir is connected to the main mercury reservoir through a solenoid operated valve. When the 110-volt solenoid is energized a glass rod is lifted from its ground-in seat permitting mercury to flow by gravity into the supply reservoir.

Automatic Controls. In the design of the control circuit (detailed in Figure 2) the following requirements were established: (1) The level of mercury in the still pot should be maintained constant during distillation. (2) Provision should be made for automatic shut-off of the pot heater and solenoid valve in case of loss of vacuum, breakage, or exhaustion of the mercury supply.

Capacity Relay. The schematic diagram of the capacity operated relay is shown as part of Figure 2. The capacity pickup consists of a piece of tinfoil approximately 1 × 2 cm. which is fixed to the outside of the still pot with its lower edge at the widest diameter of the still pot. As the mercury level in the still pot recedes from the capacity pickup, the capacity to ground decreases, causing the relay to drop out. This energizes the solenoid, opens the valve, and permits mercury to flow into the supply reservoir until the level again comes up to the pickup.

The sensitivity of control over the mercury level can be adjusted by varying C_1. At least as much of the circuit of Figure 2 as is shown enclosed by dotted lines should be shielded, and all leads in the grid circuit of the 6B8 tube should be kept as short as possible.

Heater Relay. The operation of the heater relay, also a part of Figure 2, is controlled by three probes (*A*, *B*, and *C*, Figure 1) which are positioned in the supply reservoir. During normal operation of the still, probes *A* and *B* extend below the surface of mercury, and probe *C* is above the surface. The relative heights of the probes are readily adjustable.

In order for the heater relay to turn on current to the heating mantle, electrical contact through mercury must be made between probes *A* and *B*, and probe *C* must be an open circuit. When the reset button (*SW2*) is momentarily depressed the heater relay will then pull in. If at any time electrical contact is broken between probes *A* and *B*, or contact is made with probe *C*, the heater relay will drop out.

Included in the heater circuit is the 6-volt a.-c. relay (*Re3*) which opens the circuit to the solenoid whenever the heater is off. The relay coil is shunted by a 6-ohm, 25-watt rheostat (*R12*), which can be adjusted to provide a 6-volt drop in potential across the relay coil depending upon the voltage setting of the Variac.

[1] Jelinek, H. N., C. F. Huber, and M. J. Astle, J. Chem. Educ., 26, 597–8 (1949).

This adjustment is quite easily made, as the resistance is merely increased from a minimum value until the relay pulls in.

Operation of the Still. To begin operation of the still, some mercury is admitted to the supply reservoir and the delivery tip is immersed in mercury. An oil vacuum pump is connected to the vacuum outlet and the still evacuated. The stopcock is then closed. When completely evacuated the difference in mercury level between the supply reservoir and still pot is equal to the barometric pressure. During distillation this distance is a few millimeters less because of the pressure of mercury vapor.

The level of mercury in the still pot is then adjusted to approximately the operating point of the capacity relay. This should be at a point where the still pot is about half full, if the capacity pickup is positioned properly with its lower edge at the widest diameter of the still pot. The mercury level in the still pot can be adjusted either by adding mercury to the supply reservoir, or drawing mercury from it through the stopcock.

The probes in the supply reservoir are then positioned. Probes *A* and *B* should be about 1 to 1.5 cm. below the mercury surface and probe *C* should be about 2 to 2.5 cm. above the surface.

The relays are then turned on (*SW1* closed), and the Variac turned on and set for the proper operating voltage. For the still shown, using a 125-ml. size heating mantle, the normal operating voltage is from 60 to 75 volts. After allowing at least 30 seconds for the tubes to warm up, the reset button (*SW2*) is momentarily closed. The still will now operate continuously until the mercury in the main reservoir is exhausted.

Operational Details. In designing the control unit an attempt was made to provide for all possible malfunctions that could normally be expected to occur, and prevent any damage to the heating mantle or spillage of mercury through overflow. In the event of sudden or gradual loss of vacuum in the still, the capacity relay tends to maintain the mercury at the proper level in the still pot. The mercury level in the supply reservoir will therefore rise until probe *C* is contacted. This drops out the heater relay shutting off the heater. The 6-volt relay in the heater circuit also drops out thus breaking the circuit to the solenoid. The solenoid valve must therefore remain closed.

In the event of exhaustion of the mercury supply or sticking of the solenoid valve, the mercury level in the still pot and supply reservoir will fall, as distillation proceeds, until probes *A* and *B* break contact. This shuts off the heater and solenoid as described above. If, due to gradual leakage or changes in atmospheric pressure, probes *A* and *B* are brought into electrical contact again, the heater will not start again unless the reset button (*SW2*) is momentarily closed.

In the event of power failure, both relays drop out and the unit will not start again by itself. In case of tube failure in the heater relay the effect is the same as powerfailure.

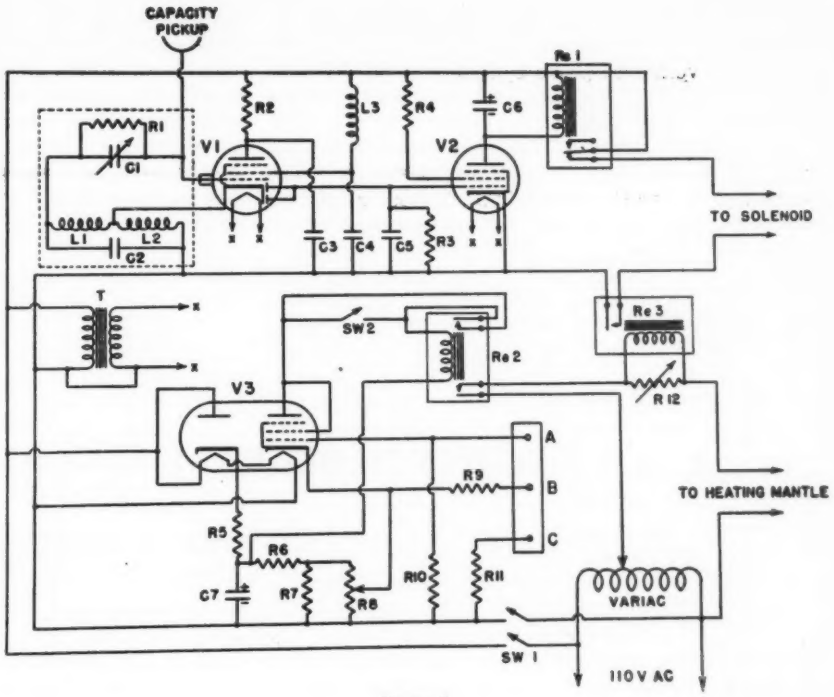


Figure 2

List of Parts in Figure 2

R1, R10. 3.3 megohms, 1/2 watt	*C5*.....0.1 mfd. paper, 400 volt
R2, R4...4700 ohms, 1/2 watt	*C6*.....8 mfd. electrolytic, 150 volt
R3.....1 megohm, 1/2 watt	*C7*.....16 mfd. electrolytic, 150 volt
R5.....200 ohms, 2 watt	*L1, L2*...1 mh RF choke
R6.....10,000 ohms, 1 watt	*L3*.....80 mh choke
R7 .....4700 ohms, 1 watt	*T*.....6.3-volt filament transformer
R8.....5000-ohm potentiometer, wire-wound	*Re1*.....Single pole plate circuit relay
R9.....0.47 megohm 1/2 watt	*Re2*.....Double pole plate circuit relay
R11.....47,000 ohms, 1/2 watt	*Re3*.....6-volt a.-c. relay
R12.....6-ohm, 25-watt rheostat	*SW1*.....Double pole toggle switch
C1.....3–30 mmf., variable trimmer condenser	*SW2*.....Push button switch, normally open
C2.....100 mmf. mica	*V1*.....6B8
C3, C4...0.01 mfd. paper, 400 volt	*V2*.....6V6GT
	V3.....117L7/M7GT

In case of tube failure in the capacity relay the solenoid valve will open allowing mercury to flow into the supply reservoir until probe *C* is contacted. The circuit to the solenoid will then be broken and the valve will close.

The still has an output of 1.2 pounds of mercury per hour when operated at 70 volts on the heater. The main supply reservoir can safely hold about 30 pounds of mercury above the outlet, which is sufficient for approximately 25 hours of operation. The output of the still is limited mainly by the cooling capacity of the air condenser. If it were made slightly larger the capacity could no doubt be increased somewhat. No difficulty with bumping has occurred, even when operating at peak capacity. If the level in the still pot becomes very low bumping will occur, however. The upper part of the still pot should be thermally insulated with asbestos.

SCIENCE IN GENERAL EDUCATION

JOHN B. ENTRIKIN
Centenary College, Shreveport, Louisiana

THE scientist is, or should be, very much interested in the public's attitude toward science as a whole. Do the editorial writers, the news-casters, the law-makers, the administrators of governmental bureaus, the school teachers, the preachers, and all the others who are largely responsible for formulating public opinion and regulating the lives of all of us have any real understanding of science—what it is and what it is not, what it can do and what it cannot do, how it has developed, and how it functions? Any affirmative answer to that question would have to be very faintly whispered at best. Yet it seems highly desirable, if not essential, that such an understanding of and appreciation for science as a whole should exist. It is estimated that not more than 1 per cent of Americans are scientists. We need to make at least the leaders of the 99 per cent literate as regards science. The problem has many facets. This article will deal with only one of them, namely, the teaching of science in college to those students whose major interest lies outside of the field of science. It should be remembered that it is from this group that most of society's leaders will come.

What type of science courses should the nonscience students take? This is not a new question, but the interest in its possible answers is now quite high. Perhaps a brief consideration of the history of science teaching will help us get our bearings. Scientists seem to have forgotten that during the eighteenth and nineteenth centuries, scientists were "natural philosophers" who tried to inform the general public regarding their work—the "how" as well as the results. It was during the last part of the nineteenth century that science began to be recognized as a more or less loose federation of highly specialized subdivisions. Most scientists quit trying to interpret science to the laymen. They even quit trying to explain their own specialty to scientists in other fields. As a result, the teaching of science became completely departmentalized and the main objective of each department seemed to be to produce other specialists in that field. The decided majority of college students—namely, those not preparing for careers in a scientific field—took as their required science course one primarily designed for those who expected to major in the department.

By the end of World War I science teachers and administrators in many colleges had concluded that the nonscience students were not getting a broad enough concept of science from the orthodox, first-year, departmental courses. This conviction led to the inauguration of the survey courses in science that were rather popular for a decade or more. Many colleges offered a course that surveyed the fields of physics, chemistry, geology, and frequently some astronomy and mathematics; and another course that surveyed the biological fields. These courses were fact-centered and each of the departments involved tried to see to it that all of the "essential facts" of that field were covered in the course. Generally, no laboratory work was included in such courses. As conceived, such courses were extensive rather than intensive, and fact-centered rather than student-centered. As taught, most of the courses became superficial.

After a few years most colleges that had introduced survey courses either abandoned them or modified them materially. These efforts to find something better than the survey courses without going back to the traditional departmental courses led several colleges and universities to strike out on new trails in the field of science education.

The writer was given a grant to allow him to confer

with teachers in more than fifty colleges and universities where some of these new approaches are being tried and to attend the workshop on "Science in General Education" at Harvard University, July 10–13, 1950.[1] Based on the information thus gained and also on studies made of pertinent published material,[2] the writer has gathered some data and reached some tentative conclusions that may be of some interest to others who are interested in what science teachers should be doing for the nonscience students. It is fully recognized that this study is not inclusive of all the colleges that are doing outstanding work in this field, but it is believed that the institutions are representative.

We are not concerned here with the curriculum of those students who plan to specialize in science in any of its areas, but rather with the science education of the majority of students—what we may call general education for citizenship. The first question to arise is, what should be the aims and objectives of these courses in science? Professor M. Rogers of Princeton University has answered:[3] "In general education we need not start the training of professional students...we need not try to equip everyone with a lot of scientific knowledge...but we do need to give an understanding of science and its contributions to the intellectual, spiritual and physical aspects of our lives." Professor Wesley N. Tiffney of Boston University has stated:[4] "Science is so vital a part of our national and international life and culture that it is highly desirable for today's citizens to have an appreciation of its achievements of an intellectual and philosophical nature and its utilitarian applications, as well as an understanding of its dangers and limitations." In describing a course, Professor Leona Sundquist of Western Washington College of Education writes:[5] "Emphasis is placed on the orientation of the student to the problems of everyday life and to the facts, concepts, principles, and techniques of science which can serve as tools to develop more efficient ways of thinking and living." Dr. Earl J. McGrath, U. S. Commissioner of Education, summarizes his findings as follows:[6] "Though many institutions have a list of eight or ten objectives, they can be classified under a relatively few heads, the most common of which is the cultivation of the scientific method and the ability to use it.... Another common objective has to do with the facts of science.... The third most common objective is concerned with the impact of science on modern life.... A few institutions, but an increasing number, have as a fourth objective acquainting the student with the historical development of science to reveal how the cooperative efforts of succeeding generations of scientists have been responsible for our present knowledge and to indicate the evolution of scientific thought."

A second question is, what should the course content be? The aims and objectives of a course largely determine its content. For the traditional beginning courses in any science, the objective, whether acknowledged or not, is to prepare the student to pursue adequately further work in that science. It is logical, therefore, that the contents of such a course be cumulatively factual and specialized. Desirable objectives for a science course for general education would certainly necessitate a serious reconsideration of the subject matter to be presented. Important as the subject matter is, however, it is probable that the attitude and viewpoint of the teacher and the awareness he has of the objectives of general education will determine the worth of the course.

Fortunately, there is no "one and only" way by which science can be successfully taught to nonscience students. The courses that are being offered by the colleges and universities that are trying to meet this need differ considerably. Even so, they fall into one of three general types: (1) the single-subject course, (2) the physical science and biological science courses, and (3) the courses in science as a whole. Each type has its vigorous proponents. Of the sixty-two colleges and universities surveyed by the author, seven have only the traditional courses in science, eighteen have single-subject courses designed for general education, thirty-five have integrated courses in the physical sciences and, or, the biological sciences, and two have integrated courses in the whole field of science.

As a chemist, the author is disturbed by the fact that chemistry does not seem to be making its proportionate contribution to these programs of science in general education. This appeared to be true in many of the institutions visited and the conclusion was strengthened by many of the comments that were made at the Harvard workshop. It is not desirable that, by default on the part of its own devotees, any branch of science fail to be represented when science is presented to the nonscience students who will largely determine the "public opinion" of the future. This argument holds whether the single-subject or the integrated course is to be presented. In the case of several schools visited, no chemistry course for general education was offered even though several other departments had such courses. In the case of courses in the physical sciences, it was frequently found that few, if any, chemistry teachers were actively participating in planning or presenting the integrated course. It is not to be assumed that we are arguing for a "spot in the sun" for chemistry in such programs, but rather that its justified usefulness not be neglected.

No attempt will be made here to discuss the arguments for the different types of courses or to list their possible contents and methodology. Interested persons will find these topics rather fully discussed in the various chapters of the book edited by Earl J. McGrath

[1] The grant was made by the Carnegie Foundation for the Advancement of Teaching and Centenary College.

[2] Particularly EARL J. MCGRATH, "Science in General Education," Wm. C. Brown Co., Dubuque, Iowa, 1948; and the *Journal of General Education*, published by the University of Chicago.

[3] MCGRATH, E. J., *Op. cit.*, pp. 1–2.

[4] *Ibid.*, pp. 171–2.

[5] *Ibid.*, p. 198.

[6] *Ibid.*, pp. 386–9.

which has been referred to previously. The chapters are written by professors in different schools and with differing points of view, but with the same over-all purposes in mind, namely: how can the hours allocated to the natural science division be best utilized with the nonscience students?

Three problems are outstanding in the successful execution of a science program in general education. These are: (1) the securing of good teachers for the courses, (2) the question of laboratory work, and (3) methods for the evaluation of the results. The really critical problem seems to be the matter of teachers. The highly specialized training in a specific science required for the doctorate does not, in general, tend to prepare a teacher adequately to teach a broadly conceived course, even in a single subject, to say nothing of an integrated course in science. A prerequisite to the further development in the teaching of science for general educational purposes is an adequate program for staffing the courses with teachers who are really interested in doing the job and who are at the same time broadly enough informed. A few such teachers already exist and a large number of the present science teachers could prepare themselves for this work if they were sufficiently motivated. One of the major handicaps to inducing present teachers to go into this field is that in many departments promotion depends largely on the quantity and quality of scientific research that the individual does. Work in the field of general education does not lead directly to scientific research as that term is presently conceived. Furthermore, experience has shown that the teaching of these courses takes at least 50 per cent more of the teacher's time and mental energy than a conventional course in a single subject, thus cutting into the time left for individual research. It would seem, therefore, that if an institution is to staff such courses adequately the teaching load of the staff members must be reduced below normal expectancy and the long-range program for promotions in rank must be based almost solely on teaching efficiency rather than on technical publications. Successful teaching in this area cannot be done by cast-offs who have proved to be poor teachers in a specialized department. An enthusiastic and competent teacher who has the confidence and active cooperation of his scientific colleagues and the support of the administration is essential.

As a long-range program for preparing teachers for science in general education, it would seem that provision should be made for their training in the graduate schools. Such a program would be somewhat revolutionary since the training should cover the broad field of science—its development, its methods, and its impact on man. Time would not permit more than a moderate degree of specialization in any one field and the term "research" would have to be thoughtfully interpreted. It does not seem that the mere taking of a number of traditional courses in various departments of science would be adequate. Beyond that, provision must be made for their integration and interpretation. The demand for such teachers is great at the present time. It seems very likely that the supply cannot catch up with the demand for many years to come. Many schools that are now teaching single-subject courses would change to integrated courses covering several fields of science if qualified teachers were available. The division of science into physical science and biological science would be eliminated in many institutions if the staff problem could be solved.

The problem of laboratory work for general education in science is one on which there is a great divergence of opinion. In the majority of cases some type of laboratory work is offered. In many institutions where laboratory work is not included, it is the lack of physical facilities and staff rather than lack of belief in its importance that has been the determining factor. In any event, the laboratory work for such courses must be realistically incorporated into the type of course that is being taught. It must be a definite implement used to reach the objectives set up for the course—not just "busy work" to consume the allotted time.

The problem of proper evaluation of any course has not been satisfactorily solved. There are two problems: (1) what are you trying to measure, and (2) how can it be reliably measured? In general education courses, the problems of evaluation are even more difficult than in the traditional courses because the objectives are less easily stated and the things that are to be measured are more difficult to measure. General education tries to develop in the student an insight into the assumptions underlying the scientific process, the nature of that process, the kinds of reality with which it deals, and the kinds of truth which it yields. Evaluation of the results is difficult, but those who have had considerable experience in the field definitely feel that it can be done effectively. The fact that it is a difficult job should make it all the more challenging to our ingenuity.

After having talked to approximately fifty of the professors who are now teaching such courses, one can truthfully say that they are, in the main, not only enthusiastic but in many cases evangelistic about their work. One professor remarked, "Anyone is crazy to agree to teach a general education course in science, but now that I have taught one for four years, I wouldn't give it up for anything." These men feel that they are making science meaningful to those students who do not have a professional interest in it, and who have thought of it only as a great mass of uninteresting and useless facts to be memorized en route to a college degree.

Science marches on! How can the great majority of college students be made to understand and appreciate its importance to them as individuals and to civilization? In all probability, something different from the conventional first-year courses in college science is required. Time will tell which of the presently used approaches to the problem is most productive. The more "experiments" that are intelligently tried, the more rapid the general progress should be.

FRIEDRICH WILHELM SERTURNER, THE DISCOVERER OF MORPHINE

GEORG LOCKEMANN
Mühle Hollenstedt, Hanover, Germany
(Translated by Ralph E. Oesper)

JUST as the modest facilities of a small apothecary laboratory sufficed for the brilliant Carl Wilhelm Scheele to discover various acids (tartaric, citric, malic, etc.) which occur in plants, chemistry is likewise indebted to the experimental skill of a young pharmacist for initiating the study of plant bases, *i. e.*, the alkaloids.

Friedrich Wilhelm Sertürner was born on June 19, 1783, at Neuhaus near Paderborn. His father, Joseph Serdiner (the spelling of the name has changed several times), an Austrian immigrant, was in the service of Friedrich Wilhelm, Prince-Bishop of Paderborn and Hildesheim, as engineer and building inspector. He had the additional duty of carrying on alchemical experiments, which reportedly led to the transfer of whole piles of silver dishes from the episcopal household to the melting pot. The father's early death (1798) was soon followed by that of his employer, who had been the boy's godfather and patron. Consequently, at 15, young Sertürner had to abandon his intention to follow his father's profession. Because of his scientific leanings, he—as once did Scheele in a similar situation—decided to become a pharmacist.

In the fall of 1799 he became apprentice to the court apothecary, F. A. Cramer, in the Adlerapotheke at Paderborn. After four years of training, he passed the assistant's examination with distinction, and then stayed two and a half years longer with his excellent teacher, to whom he publicly acknowledged his respect and gratitude as late as 1826 because "with an expert and fatherly hand he guided me in my youth into the serious and comprehensive domain of science."

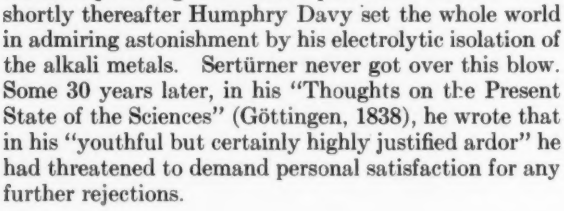
In addition to his professional duties the industrious apothecary's assistant carried out all sorts of chemical experiments (detection of benzoic acid in fennel water, of saltpeter in beets, etc.). He soon devoted himself to the seductive task of searching out the active constituent of poppy juice. Success came quickly; he found that the juice contained a new acid, which he named "mohnsaüre" or "mekonsaüre" (*Mohn*, German, and *mekon*, Greek = poppy). However, trials on dogs showed the material to be inactive. Addition of ammonia produced a precipitate, which he obtained in crystalline form from hot alcohol solution. He found that this product was a base since it turned violet juice green. Animal experiments proved that it was the actual "*principium somniferum.*" This finding was all the more surprising because it had been generally assumed that the stupefying action of opium was due to a resinous or volatile ingredient.

Friedrich Wilhelm Sertürner

Sertürner communicated his discovery to Johann Bartholomäus Trommsdorf, the leading authority in the field of pharmacy, who was professor at Erfurt. The latter published it in his *Journal der Pharmazie* [**13**, 127 (1805)]. A more detailed account appeared in the same periodical the next year [**14**, 47 (1806)]. In a postscript, Sertürner stated that not until his experiments were completed had he learned of the publication in the *Annales de Chimie* [**45**, 257 (1804)] in which the French apothecary Derosne had reported on similar experiments. However, the latter had thought the precipitate was a salt and not an organic base, and he ascribed the effect on violet juice to adhering alkali. Sertürner made specific mention of this error, but his paper received almost no attention.

In the meantime (Easter, 1806), Sertürner had moved from Paderborn to Einbeck, where he had accepted a post as assistant to Hink, the Council apothe-

cary. Here, at first, he took up other scientific problems. He also prepared an essay on galvanism which he entered in a contest sponsored by the French "Institut National," and among other experiments worked with pyrophori, which he compounded from lampblack, sulfur, and potassium salts. This work brought him to the belief that the alkalies are not elementary in nature, as was still generally assumed, but instead are made up of oxygen and inflammable elements "similar to hydrogen." This notion seemed so outlandish to the editor, Professor Gehlen, of Munich, that he hesitated to publish Sertürner's paper in his *Journal der Chemie* for fear of being ridiculed. This rejection of his manuscript brought Sertürner particular distress when shortly thereafter Humphry Davy set the whole world in admiring astonishment by his electrolytic isolation of the alkali metals. Sertürner never got over this blow. Some 30 years later, in his "Thoughts on the Present State of the Sciences" (Göttingen, 1838), he wrote that in his "youthful but certainly highly justified ardor" he had threatened to demand personal satisfaction for any further rejections.



"Hochzeitshaus" in Hameln Where Sertürner Had His Apothecary Shop

The house to the left and the church in the rear were destroyed by bombs during World War II. Sertürner's son moved the apothecary across the street, where it still exists.

In 1809 the authorities of the Westphalian kingdom, which had newly been created for Napoleon's brother Jerome, granted Sertürner a license to open his own apothecary at Einbeck. Though busy with a wide variety of studies he now came back to the opium studies because some had questioned the physiological activity of the crystalline material, which he as well as Derosne had prepared. He pointed out that because of its water insolubility, this material as such could not be active; to render it effective, it is necessary to use it in alcohol or, as a salifiable base, to dissolve it in acid. To further the national economy, he recommended the cultivation of the poppy on a larger scale, since such plantings could yield not only the active ingredient of opium but also poppy seed oil which would be valuable as a substitute for the expensive imported olive oil.

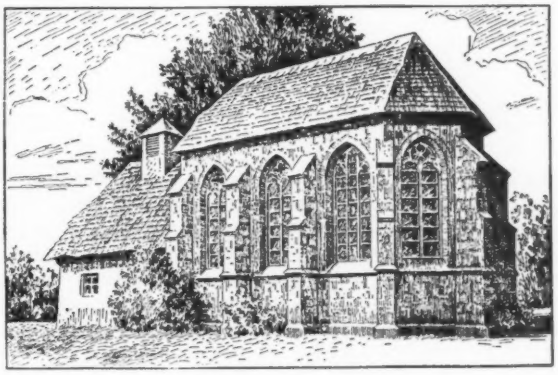
After astonishingly varied pursuits, including especially an extensive series of experiments for the improvement of ordnance (it was the period of the War of Liberation), Sertürner, from 1815 on, again took up his opium studies in earnest. He supplemented his previous investigations and showed that the active ingredient contains nitrogen along with carbon, hydrogen, and oxygen. He named it "morphium" after the god of dreams, and since it was a salt-forming base, he included it in the series of alkalies, placing it after ammonia. He tried its physiological action on himself and three young volunteers. Over a period of 45 minutes they took three doses of $^1/_2$ grain (about 30 mg.) of morphium dissolved in alcohol and diluted with water, *i. e.*, a total of about 0.1 g. This exceeds the present maximum daily dose of 0.1 g. morphine hydrochloride. The action was correspondingly great. Sertürner, already half unconscious, tried to combat the toxic manifestations by administering massive doses of an emetic. It was several days before all the evil consequences of this courageous experiment were overcome. Sertürner found that he could tolerate 5 grains (about 0.3 g.) of his meconic acid without noticing any particular action.

Sertürner's paper [Gilbert's *Annalen der Physik*, **55**, 56 (1817)] attracted widespread attention. As early as May 4, 1817, he was named ordinary member of the "Societät für die gesamte Mineralogie" at Jena, whose president was no less a personage than Goethe. The University of Jena honored him with a Ph.D. *in absentia*. His discovery was recognized abroad also. Gay-Lussac inserted a French translation of Sertürner's paper in the *Annales de Chimie* [**5**, 12 (1817)] and expressed his astonishment that this "decouverte extrément importante" had gone unnoticed for so long both in Germany and other countries. But despite this acclaim by one of the most distinguished French scientists Sertürner was attacked several years later by another eminent Frenchman. Vauquelin [Gilbert's *Ann. d. Phys.*, **65**, 381 (1820)] declared that Sertürner's paper was a plagiarism of a study by Seguin, which the latter had handed in to the Academie in December 1804, but which did not appear in the *Annales de Chimie* until ten years later [**92**, 225 (1814)]. Seguin had likewise shown the presence of meconic acid in opium juice and had prepared the crystalline precipitate, but he had no more recognized the innate basic nature of the precipitate than had his countryman, Derosne. Consequently, Sertürner's satisfaction was all the greater when, in 1831, he was awarded the annual prize of 2000 francs by the Institut de France, which was notoriously conscientious with respect to nationalistic questions. The accompanying citation, composed by Cuvier, the Permanent Secretary of the Academie, read in part: "pour avoire reconnu la nature alcaline de la morphine, et avoire ainsi ouvert une voie, qui a produit des grandes decouvertes medicales." In this connection it should be noted that this same Institut four years before had awarded a prize of 10,000 francs to Pelletier because of his success, using Sertürner's process, in isolating the active ingredient of Cinchona bark.

Sertürner himself was fully aware of the great significance of his discovery. He predicted that other basic plant materials would be found. In fact as early as 1817, Robiquet showed that Derosne's crystalline precipitate contained a second active ingredient, which he called "narcotine." The list was soon extended by the discovery of strychnine, brucine, veratrine, etc., so that twelve of these active substances were known by the end of 1820 and about 30 by 1856. The generic term "alkaloid" for these alkaline plant substances was proposed in 1818 by W. Meissner.

Sertürner's personal circumstances had undergone great changes in the meantime. The Westphalian kingdom had, of course, gone down along with Napoleon and Sertürner was compelled by the restored Hanoverian régime to give up his pharmacy. Since he had no success in finding another suitable sphere of activity, he devoted himself predominantly to writing. He compiled an extensive work, which he dedicated to the memory of Lavoisier, Newton, Mayow, and Richter. This "System der chimischen Physik oder Entdeckungen und Betrachtungen im Gebiete der Chimie und Physik" was published in two volumes at Göttingen in 1820 and 1822. Finally, after years of negotation, he was given permission in 1820 by the Hanoverian authorities to take over the Ratsapotheke in Hameln. He was now able to marry, and he lived in the "Hochzeitshaus," a famous renaissance structure, for about 20 years. He became one of the prominent citizens of this famous old city, which is best known as the scene of the Pied Piper of Hamelin. He died there, a victim of the grippe, on February 20, 1841. His body was taken to Einbeck and laid to rest in the church in the vault of the patrician family Wernher, from whom his mother-in-law was descended. A simple stone was erected there in 1917 by the Apothekerschaft.

Sertürner's first scientific achievement, which came when he was only 22, remained his greatest success. With his many-sided interests, he was always getting involved in the most varied problems, whose actual solution was beyond him. Since, at first, he was not given proper credit for his morphine discovery, he felt later whenever his views were not accepted that his critics were actuated solely by jealousy and envy. Consequently, he persuaded himself more and more that he was an unappreciated genius. With his theory of ether formation and other chemical processes he experienced the tragic fate, not of being disputed, but of being passed over in silence. Since his experiences with the editors of several scientific periodicals had been none too happy, he founded a journal of his own with the high-sounding title: "Annalen für das Universelsystem der Elemente." It breathed its last after four years (1826–29). Its columns contained papers on such things as the remarkable vital element "zoon," the cold nature of sunlight, a wide variety of medicaments, the science of ordnance, and many other topics. All this chaff threatened to bury his really good scientific achievement. However, it remains to his immortal glory that he brought suffering mankind a wondrous magic potion that eases pain, and thus also opened the way to many other extremely valuable vegetable materials.



Chapel at Einbeck Where Sertürner Is Buried

His clear independent insight into puzzling natural events was shown in still another way. Asiatic cholera broke out in Europe in 1830 and Germany was invaded by this dread disease. In his "Dringende Aufforderung an das deutsche Vaterland in Beziehung der orientalishen Brechruhr" (Göttingen, 1831) Sertürner developed the idea that this disease is due to extremely small organisms, which in size are to a mite as the latter is to a prehistoric giant. He also gives directions for disinfecting water in which the disease carriers live, and demanded that only boiled water be used. Even though, in earlier times the notion had been occasionally advanced that minute creatures are the cause of infectious diseases, such ideas had been entirely abandoned at the beginning of the nineteenth century, and the "miasma theory" was accepted without question. Independent of the views that prevailed in scientific circles of his day, Sertürner had been advocating his view for about ten years before the Göttingen pathologist Jacob Henle developed the theory of "*contagium vivum*," and a half century before Robert Koch succeeded in bringing experimental proof of the correctness of this view.

ERRATUM

In the March issue the captions of three portraits have been mixed. They are von Jacquin (page 138), Werner (page 140), and Haüy (page 141). These portraits should be captioned Abraham Gottlob Werner, René Just Haüy, and Joseph Franz Freiherr von Jacquin, respectively.

Proceedings of the

PACIFIC SOUTHWEST ASSOCIATION OF CHEMISTRY TEACHERS

THE USE OF LANTERN SLIDES IN UNDERGRADUATE ORGANIC CHEMISTRY[1]

NORMAN KHARASCH
University of Southern California, Los Angeles 7, California

Lectures in organic chemistry have a considerable tendency to become a twofold race against time. The instructor, with a prescribed amount of text in mind, busily engages himself in putting a wealth of detail on the blackboard; while the student, burdened by the pressure of examinations to follow, madly attempts to record it in his notes before the material presented is erased. The oft-repeated lament of the student, "He puts it on with one hand and erases it with the other," is not entirely fictitious.

It is certainly true that by careful selection of material, by lucid and stimulating presentation, and by diligent preparation on the part of the student as well as of the instructor, the good teacher can and does largely avoid the above difficulty; and some, miraculously enough, can still find time within the lecture period to answer questions and to present some helpful demonstrations. But because of the extent of organic chemistry, and the need to develop the subject in sufficient detail to assure a realistic attitude to the work, the delivery of lectures in organic chemistry in a satisfactory manner is a challenging task which calls for the utmost of skill on the part of the instructor.

In an attempt to increase the effectiveness of his own lectures to undergraduate classes in organic chemistry, the author has experimented for the past three years in presenting the material from lantern slides. The blackboard is used quite freely, but only as a secondary help—to clarify now and again the discussion relating to the slides, and to give additional instruction as the needs of the moment may demand. The execution of this experiment has been quite enjoyable; and judging by the response and performance of hundreds of students during several terms, the method seems to commend itself for serious consideration.

On occasions when this development has been discussed with colleagues, there appeared to be good agreement on the point that slides could be very effective for lecture instruction; but contradicting this, and equally definite, was the feeling that there are certain difficulties which militate against the general use of slides in the classroom. Let us enumerate some of these objections and comment upon them in view of our own experience.

A most frequent observation is that the preparation of slides is time-consuming, expensive, and accompanied by a bothersome storage problem. Furthermore, that once prepared, the slides will be used without revision for years, leading to a continually less effective course from term to term.

In our experience, none of these factors need be serious. The majority of the slides used are made on the commercially available "Radio-Mats." Exclusive of glass covers, which may be used repeatedly as slides are revised (retail price about eight cents a set), the cost of making a suitable cellophane mat slide is only two or three cents per mat. As to the time required to make such a slide, it can be done as quickly as one wishes to write, draw, or type the material onto the mat. Thus, a very adequate set of cellophane mat slides, which may be kept for years or revised at will, can be made at low cost and with little or no more effort than is required to prepare a set of hand-written notes.[2]

Although the acquisition of some black and white, as well as some colored, slides is advisable, it is true that the cost of the photographic slides is higher, especially if they are sent out for processing. However, the photographic slides are likely to be suitable for longer continued use; and some of these can, if desired, be effectively prepared in one's own laboratory at moderate

[1] Summary of a lecture presented before Symposia arranged by the Pacific Southwest Association of Chemistry Teachers, San Francisco, Nov. 4, 1949, and Los Angeles, Nov. 4, 1950.

[2] In writing or drawing on the Radio-mats, a helpful technique is to clip a square of the waxed paper and one of cellophane onto a glass plate, and to write directly onto the cellophane mat with a fine ball-point stylus.

Dr. Karol J. Mysels has suggested an effective way of making slides quickly on frosted cellulose-acetate film of suitable thickness (0.0075 in.). The mats may be inserted between waxed paper, and typed; or written onto with a colored or soft black pencil. The technique is especially useful for tracing small drawings or charts from books or journals directly onto the transparent cellulose-acetate mat. Frosted glass plates may, of course, also be used for the latter purpose.

cost. Hand-coloring of photographic slides is somewhat of a lost art, but makes a fascinating hobby for those who may choose to pursue it.

As to revision of slides and the effect of this on the course: lethargy and lack of time may indeed result in failure to make the needed changes from term to term. But are these not the same factors which would retard the proper development of a course of lectures presented in any other fashion? And concerning the storage problem: we have found that convenient boxes for cataloguing and storing slides are available at relatively low cost. In the type of box selected, the storage of 75 slides requires only $3^1/_2$ in. of book-shelf space.

A second point, often mentioned, concerns the need to darken the lecture room so that students are unable to take notes; and worse, "They go to sleep."

A fairly modern projector with a thousand-watt bulb, used in conjunction with a "daylight" screen, gives sufficient brilliancy so that the image is perfectly clear, even in a large room with nearly normal daylight illumination. In some lecture halls, especially when showing photographic slides, it is desirable to reduce the illumination in the vicinity of the screen; but certainly there is no general problem in reserving ample illumination for taking notes. And with regard to keeping the audience awake: this is a problem which is not unique to lectures in which slides are used. In addition to the usual techniques which make for good audience contact, we have found that the well-timed projection of a cartoon slide or of a photographic slide of unusual interest allows for a brief diversion which proves quite effective.

It has also been suggested that the instructor is freed of the labor required to put "all the stuff" on the board, and will give much more material than the class can possibly keep up with.

This is a very common fault of lecturers who use slides. But cannot the remedy be simply effected by taking the difficulty into account?

A fourth but minor objection is the comment that the use of slides calls for the service of a lantern operator.

This problem is easily overcome. If one avoids the pitfall of going too fast and of using too many slides, any student can operate the lantern with a minimum of effort and without losing any of the lecture context.

GUIDE NOTES

Soon after the lantern-slide method of presentation was adopted, it became obvious that a set of guide notes to the lectures for student use, might be very helpful. The preparation of such a set of notes was undertaken with some trepidation. Not only would the proper development of the notes require considerable effort; but of greater concern was the possibility that their availability might reduce student incentive for attendance at lectures. The format chosen for the notes was such as to lend itself to the presentation of lectures from slides. In general, a page of the notes corresponds to a single lecture, and is accompanied by eight to ten slides, and the development of such *unit lectures*—each dealing with a stated topic—has been a critical feature in the growth of the author's course. This purpose, to present each lecture as a suitable unit unto itself, has been well fostered by the preparation of the guide notes and by the manner in which they are used. A brief demonstration is also planned for each lecture, and a short period of discussion is often allowed at the close of the hour.

The notes and the exercises which are supplied to the student are definitely not intended to supplant the textbook or the original literature; nor are they, by any means, graphic facsimiles of the oral lecture. Indeed they may be so planned and used as to assure that the student will use his book constantly while solving problems and reviewing his notes. By inclusion of appropriate exercises and references, the occasional reading of original articles is also encouraged.

The lectures and demonstrations will mean most to the student only if he prepares for them carefully, both before and after the lecture period. No specific recommendations need be made at this point, since this is a task which is quite entirely the responsibility of the student. We have, however, observed with interest the various study techniques which our students have developed to fit the present organization of the lectures; and we have been particularly impressed with the manner in which many of them have incorporated the printed notes with their own records of exercises and of class and reading notes into a quite comprehensive and useful compendium. The students still take a considerable number of notes during the lecture, but these are marginal ones—on critical points, or on new material over and above that already available for them in the printed notes or textbook.

Although we are of the opinion that no lowering of standards, whatever, has been incurred by adopting the present mode of lecture presentation, we are well aware of the criticisms which may be levied against the use of such class notes as were described above. It is our belief, however, that the difficulties are not inherent in the fact that the notes are made available, but rather in the possibility that the major requirement of the course may become the rote memorization of such a set of notes, rather than their critical use toward a broader objective. Our experience strongly implies that the proper use of the notes and slides permits the instructor to conduct his lectures in a manner which impels the students to seek the truly important aspects of the course.

In a good presentation of any subject, the lecturer must be free to observe his audience and to be with them; and he must permit his listeners to formulate questions in their minds. In this connection, the old rule for effective teaching, "First tell them what you are going to tell them about; then tell it to them; and then tell them that you have told it to them," can be a useful guide to good results if it is done lightly and with imagination. Visual aids as such certainly cannot lend the inspiration for a good lecture; but properly and simply used, they can free the lecturer to do his best work.

Report of the

NEW ENGLAND ASSOCIATION OF CHEMISTRY TEACHERS

ELECTROCHEMISTRY IN THE FRESHMAN COURSE[1]

WILLIAM B. MELDRUM
Haverford College, Haverford, Pennsylvania

THE object in presenting this paper to this group of high-school and college teachers is twofold: first, to call attention to one type of college chemistry course in which credit is allowed for work done in the subject in high school; and, second, to emphasize the importance of electrochemical concepts in the freshman chemistry course. In doing this the author finds it convenient to utilize the experience accumulated in the more than quarter century of his own teaching.

For nearly twenty years a system has been in operation at Haverford College whereby students, most of them freshmen, are admitted directly to a second course in college chemistry on the basis of their preparatory school course. They are carefully selected and, on the average, about 25 per cent of those who attempt to elect the course are "persuaded" not to take it. Judgment of a boy's fitness to enter this second course is based upon several criteria of which the chief are: first, his rating in the College Entrance Examination Board Achievement Test in chemistry; second, his rating in the school course; and, third, the general rating of the school from which he comes as indicated by the degree of success in our course of former graduates of the same school. If his course in school has been of standard length and coverage, with suitable laboratory experience, and if his College Board rating in chemistry is 550 or over, then he is usually admitted. If he has not taken the College Board test in chemistry, then his ratings in mathematics and physics are examined. Such records as these are made available to the Department by the college office. Those students who have not had chemistry in high school and those whose preparation is considered inadequate for creditable performance in the course may enter the elementary course, and then, if they wish to pursue the study of chemistry further, they may elect the same second course in the following year. Formerly, the Chemistry Department could "demote" to the elementary course those students who showed within the first five or six weeks of the term that their preparation was inadequate or their habits of study unsuitable to ensure creditable performance. This desirable privilege was, however, ended in the postwar reorganization of the college curriculum and its termination has necessitated more careful screening in order to avoid the admission of students who are unprepared to do good work in the course.

A student thus admitted to the second course is enabled to proceed more rapidly into the more specialized courses in chemistry and is required to repeat relatively little of what he has already learned in high school. The results have been uniformly satisfactory. In the first place, the psychological effect on the student is good; thus admitted to an *advanced* course he feels that he is put on his mettle and tries to do good work; the depressing and relaxing effect of spending time in repetition of what he has already studied in school is largely eliminated and, definitely, he finds the work more interesting. Our experience shows conclusively that we are not being over optimistic in expecting the abler and more alert students to carry over and apply in the college course what he has had the year before, or the second year, before, in high school, especially if that material has been well taught. Also, the subsequent success of these students in graduate schools and in positions in industrial chemistry would indicate that they are not handicapped by having this type of a general chemistry program.

This second course is labeled "Inorganic Chemistry and Qualitative Analysis." Systematic qualitative analysis using semimicro methods chiefly comprises the laboratory work. The classwork emphasizes the *principles* of inorganic chemistry: "the fundamental chemical theory, reaction velocity and equilibrium, solutions of nonelectrolytes and electrolytes, the ionic theory and its applications to analytical processes and electrolytic phenomena, the periodic law, radiations,

[1] Presented at the Twelfth Summer Conference of the New England Association of Chemistry Teachers at the University of Connecticut, Storrs, Connecticut, August 22, 1950.

and the theories of atomic and molecular structure and their applications in chemistry."[2] With the aim of setting at rest the fears of some teachers that principles are overemphasized and the learning of the facts of inorganic chemistry neglected, two things may be pointed out: first, that the discussion of *principles* as here listed involves inevitably the use of many facts as illustrative material; and, second, that prospective chemists, to whom solely the learning of such facts can be of real importance, have the opportunity of expanding their factual store in a senior course in advanced inorganic chemistry and in their review for the Major examination at the end of the senior year. The author is in sympathy with those educationalists who hold that at the freshman level in eductation it is of greater value to develop principles than to memorize facts.

"Fundamental chemical theory" deals with the logical development of the principles leading to the establishment of equations, formulas, atomic and molecular weights, and other "tools" of the chemist. This is followed by "reaction velocity and equilibrium" where the principle of dynamic equilibrium and the principle of Le Chatelier are stressed because of their later importance in connection with ionic equilibria. The study of aqueous solutions and their colligative properties leads to the development of the ionic theory; the theory of Arrhenius is treated historically and the theory as it is today studied more thoroughly. Three fields of application of the ionic theory receive extensive treatment: ionic reactions, quantitative ionic equilibria, and electrochemistry. The remainder of the course, comprising about 40 per cent of the whole, deals with the periodic system, radiations, radioactivity, the atom, isotopes, and atomic and molecular structure.

About nine class hours are devoted to electrochemistry, the subject being studied with reference to a "typical electrolytic circuit." The various parts of the circuit are studied in turn: the nature of the current through the connecting wires; electrolytic conductance and its approximate measurement; ion velocities; electrode reactions, considered as oxidation-reduction reactions and as the transformation of electrical energy into chemical energy; Faraday's laws; galvanic cells of various types, including the lead storage cell and one kind of dry cell; standard electrode potentials. These are interesting in themselves but their importance is enhanced by studying them in relation to other chemical phenomena and properties, for example: (1) the relation of conductances of solutions of similar substances, such as the acids, to their chemical reactivities; (2) the determination of the end point in a titration by the resulting change in current, using such solution pairs as barium hydroxide and sulfuric acid, and silver sulfate and barium chloride; (3) the application of electrolysis in electrorefining, electroanalysis, electroplating and other electrochemical processes; (4) the development of a comprehensive system of deducing equivalent weights from equations based upon Faraday's second law; (5) the relation of standard electrode potentials to the reactivities of the metals and nonmetals as indicated in the reactivity series. In the last connection, it is interesting to point out the highly significant fact that a linear relationship exists between the standard electrode potentials of the metals and of the nonmetals and the heats of formation per gram equivalent of similar compounds such as the chlorides, bromides, or sulfates of the metals, or the potassium or sodium salts of the nonmetals. This can, of course, be deduced from the Gibbs-Helmholtz equation, but its chief interest to the student lies in its bearing on the nature of the process of combination of a metal with a nonmetal.

The above treatment of electrochemistry constitutes about ten per cent of the classwork but this becomes about 90 per cent if we accept the Websterian definition of electrochemistry as "the science of the relation of electricity to chemical changes." Much of what is listed under "radiations and atomic and molecular structure" and most of the material dealt with under "solutions, the ionic theory and its applications" belongs in this same category.

Two topics under this broader definition seem worthy of further discussion at this time: (1) the production of free ions in solution and (2) ionic reactions.

PRODUCTION OF FREE IONS

In aqueous solution this takes place by two processes which are essentially different:

(*a*) In general, the crystals composing salts and hydroxides, like potassium chloride and potassium hydroxide, consist of ions formed when the substance was synthesized from the elements.[3] When the crystal of potassium chloride is put in water, the water molecules, which are polar, are attracted to the positively charged potassium ion and the negatively charged chloride ion sufficiently strongly to overcome the lattice forces binding the ions in the crystal, and so solution takes place. We represent the process by the equation:

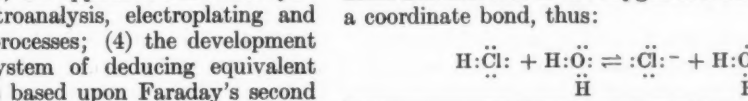
$$K^+Cl^- \rightarrow K^+ + Cl^-$$

or more simply,

$$KCl \rightarrow K^+ + Cl^-$$

In the case of most salts the change is probably complete and irreversible if solution of the substance is complete. The process is properly called *dissociation*, not *ionization*.

(*b*) If the substance is covalent in structure, like hydrogen chloride or acetic acid, ionization is now assumed to take place by reaction with the water. A proton is transferred from the solute molecule to the water molecule; the proton breaks away from the covalent bond attaching it, for example, to the chlorine atom and attaches to the oxygen of a water molecule by a coordinate bond, thus:



[2] Quoted from the Haverford College Catalogue.

[3] For a summary of the evidence see H. B. Friedman and K. E. Shuler, J. Chem. Educ., **24**, 11 (1947).

This reaction to the right is virtually complete in dilute solution.

Water in the liquid state is probably not such a simple substance as the formula H_2O implies and so the foregoing representation may be oversimplified but most chemists agree that the evidence supports this as the essential nature of the ionization process. In spite of this many authors persist in representing the change as simple dissociation, thus:

$$HCl \rightarrow H^+ + Cl^-$$

which the thinking student is almost certain to question. The practice, however, seems to be growing, for authors to give as the probably correct representation

$$HCl + H_2O \rightleftharpoons Cl^- + H_3O^+$$

and then revert to the old Arrhenius representation which is thereupon used "for the sake of convenience in writing equations." The defense is sometimes made that we do not use the hydrated forms of ions other than hydrogen, except perhaps in the modern representation of hydrolysis, and so why be so particular about the hydrogen ion? The answer to this is that we are by no means sure of the extent of hydration of many of the ions whereas the evidence for H_3O^+ from many angles seems convincing. Also, the *process of ionization* is more important pedagogically than the writing of miscellaneous equations involving other ions. In the opinion of the author, the gain in "convenience" in using H^+ instead of H_3O^+ even in writing oxidation-reduction equations, is negligible in comparison with the loss concomitant with departing from what we believe to be correct.

IONIC REACTIONS

In inorganic chemistry, but especially in qualitative analysis the student meets vast numbers of reactions involving many different reagents and substances. Equations must be written for these if the matter is to be understood. How many of these equations should the teacher expect the student to know and how can they best be learned? If we study the situation we find that by applying the concepts of the ionic theory the chaos of reactions readily reduces to order.

When dealing with aqueous solutions we find that all of the numerous reactions can be classified in four categories, as follows:

(*a*) *The Formation of a Precipitate.* The ions come together to form a substance which is nearly insoluble and thus precipitates. The lattice forces of the crystal overcome the attractive forces of the polar water molecules for the ions. This is merely the reverse of the solution process of a soluble salt. A simple example is:

$$Ag^+ + Cl^- \rightleftharpoons AgCl\downarrow$$

(*b*) *The Formation of a Weak Electrolyte.* The formation of water or of a weak acid results in the majority of these cases. If the student has learned which acids are weak and which strong he is usually in a position to predict the result in a given case. The formation of water or a weak acid from hydroxyl or the anion of the acid almost always involves the formation of a stable covalent bond and in this lies the reason for the occurrence of the reaction. This may be seen in such reactions as:

$$CH_3COO^- + H_3O^+ \rightleftharpoons CH_3COOH + H_2O$$

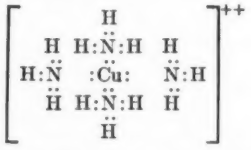
and

$$CaCO_3 + 2H_3O^+ \rightarrow Ca^{++} + H_2CO_3 + 2H_2O$$

(*c*) *The Formation of a Complex Ion.* Many complex ions are capable of formation between the more common ions tested in the laboratory and the reagents employed. A careful examination of these will show that in most cases it is probable that *a stable coordinate bond* has been formed. One of the best known cases is the reaction between cupric ion and ammonia to form the cupriammonia complex ion (tetrammine cupric ion), thus:

$$Cu^{++} + 4NH_3 \rightleftharpoons Cu(NH_3)_4^{++}$$

The composition of this complex ion is readily found from freezing point depression measurements and the evidence is reasonably conclusive that its structure is represented by the formula:



The stability of this complex ion may be ascribed to the formation of four stable coordinate bonds by which the cupric ion is attached to the nitrogens in four ammonia molecules.

(*d*) *Oxidation-Reduction Reactions.* In virtually all of the reactions of this type occurring in aqueous solution there is a transfer of electrons from one substance to another. In fact, we may say that such reactions take place because of a difference in electron affinity of the reacting substances. The function of hydrogen ion in some reactions and of water molecules in others must, of course, be carefully considered.

By the careful study of these four types of reaction the task of writing equations becomes less of a memory chore and becomes rather a matter of applying definite principles. All of the mechanisms can be ascribed to a shift of protons, electrons, or other charged particles, and in most cases this can be visualized. The mechanisms of oxidation-reduction reactions can be satisfactorily demonstrated using the familiar "reaction at a distance" apparatus not only qualitatively but in many cases quantitatively as well.

By way of summary it may be said that the author definitely favors, on the basis of many years experience, admission to a *second course* in chemistry in college if a student has had a good course in preparatory school. He recommends that such a course deal primarily with the *principles* of inorganic chemistry and that these principles are, for the most part, best understood by the

students if presented against a background of electrochemical concepts. Further, such a course, permitting, as it does, lecture demonstrations in illustration of virtually all major points, not only arouses keen interest on the part of the students but provides an excellent vehicle for the scientific method.

To the Editor:

I was very much interested in the judicious comments of your December editorial. To ask a student to "balance an equation" involves an error which must be prevented. In my class in general chemistry (elementary) I ask the students "to equationize the formulation of a reaction." An example will show what is meant by this somewhat cumbersome expression. A student is first asked to *formulate* the reaction, say, between iron and steam, and he writes:

$$Fe + H_2O \rightleftharpoons Fe_3O_4 + H_2$$

To formulate means to write the symbols and formulas of the reactants followed by the symbols and formulas of the products and separated by double (equilibrium) or single arrows, without trying to equationize (*i. e.*, to balance) anything. After that the student is asked to *equationize* the formulation and he writes:

$$3Fe + 4H_2O \rightleftharpoons Fe_3O_4 + 4H_2$$

I should like to submit to your readers these clear and consistent conventions.

RICARDO C. FERREIRA

CAIXA POSTAL, 527, RECIFE
PERNAMBUCO, BRAZIL

To the Editor:

It has been quite apparent for numerous years that only a very limited amount of inorganic chemistry is taught in our American schools. This fact was discussed at some length at the Symposium on the Place of Inorganic Chemistry in the Undergraduate Curriculum at the 116th Meeting of the American Chemical Society (September, 1949) and later summarized in THIS JOURNAL (**27**, 437–57, (1950)). I have recently taken the opportunity to satisfy my own curiosity that only a very minimum amount of inorganic chemistry is being taught in general chemistry. Since the A. C. S. Committee on Professional Training requires no inorganic chemistry on the undergraduate level, such a course is not even offered by many of our schools.

Here at Northwestern we require that all chemistry majors take a one-quarter course at the junior-senior level in descriptive inorganic chemistry. Although this course is not required of graduate students, most of them (whether or not they have had a similar course elsewhere) choose to take it, feeling that this will improve their chances of getting through the preliminary examinations. At the first meeting of this class the students (47 undergraduates, 42 graduate students) were given approximately 15 minutes to reproduce as best they could the periodic table. Although I have taught this course for the past four years, this question was never asked previously and the students had no way of anticipating it. I have found the results extremely interesting and revealing. They are given in the following table.

No. of elements correctly placed	*Undergraduates, %*	*Graduates, %*
0–9	53	33
10–19	36	35
20–29	7	18
30–39	2	7
40–51	2	5

It will be noted that over half the undergraduates and one-third of the graduate students were unable to place correctly as many as ten of the elements. Ninety per cent of the undergraduates and over two-thirds of the graduates were unable to place twenty elements correctly. Only four per cent of the undergraduates and twelve per cent of the graduates were able to place thirty or more elements correctly. Of those students who attempted to place the elements in the table, over half of the class placed over half of the elements incorrectly.

These results show that our undergraduates scored less than the graduate students from other schools. This is to be expected since many of the undergraduates have neither the interest nor ability to do graduate work, while in addition some of the graduate students have had a course in inorganic chemistry and others have been teaching assistants in general chemistry for one quarter or more. At any rate, one would expect that a good mark for any student with a major in chemistry would be the correct placing of approximately thirty elements, namely, the first two series, the alkali metals, the alkaline earths, nitrogen, oxygen, and the halogen families. Yet less than a tenth of the students were able to do this.

Undoubtedly, these findings will be criticized by some on the basis that there is no correlation between knowing the periodic table and knowing inorganic chemistry. I agree that a person can memorize the table but still know little or no inorganic chemistry. How-

ever, I am just as certain that if one has a good understanding of some of the fundamental concepts of inorganic chemistry, he can surely work out a periodic arrangement for at least twenty to thirty of the elements. For the sceptics who still feel that this is not a good measuring stick, I say try it and convince yourself. We are receiving graduate students from universities and colleges whose departments bear the stamp of approval of the A. C. S. Committee on Professional Training. These students have majored in chemistry, stand near the top percentile of their graduating classes, and have been highly recommended by their professors. Surely, it is difficult to rationalize this with the listing of the halogens as Cl, Br, F, and I or of the first series in the periodic table as Na, Cu, Al, C, S, N, and Cl.

FRED BASOLO

NORTHWESTERN UNIVERSITY
EVANSTON, ILLINOIS

To the Editor:

I have taught chemistry in a "one-horse" department for many years. (I was the horse.) Of all the courses I attempted to cover, I enjoyed teaching qualitative analysis as much as any, and I think my students shared in my enthusiasm. Therefore I have been surprised to read a steady barrage of criticism of this course during the last quarter century. One might have expected criticism that general chemistry is too general, or that quantitative analysis is too narrow, or that organic chemistry has too much to be memorized, but the facts are that qualitative analysis, which was so highly praised by its friends, has been singled out for special blame.

Several writers have observed a "trend" toward incorporating qualitative analysis in general chemistry. Most of these authors have condemned qualitative for this trend and have hinted that it is in danger of losing its place on the college curriculum. When I examine the evidence, however, I come to the opposite conclusion. There has been an unquestionable trend, but it has not been accompanied by a corresponding decrease in the number of schools offering qualitative analysis as a separate course.

Qualitative analysis when taught on the freshman level cannot maintain as high standards as when it is offered to sophomores. It is no fault of qualitative but is due to the immaturity and lack of background of the freshman students. If qualitative were to lose its place as a separate course and if it were to be relegated to the freshman year, it would lose much indeed. However, I find an element of strength even in this so-called trend. There must be something dynamic about qualitative analysis to enable it to maintain a steady march into the very citadel of general chemistry until in more than half of the schools, it has taken over the second half of the laboratory work. This something, I believe, is the *motivation that comes from solving unknowns*, and all the sciences have borrowed it from qualitative.

The experience at Monmouth College which can be verified by results from cooperative tests is that the students in general chemistry are not ready to grasp the mathematical concepts of equilibrium, mass action, solubility products, ionization constants, oxidation and reduction, ionization potentials, etc. When attempts were made to teach these in the freshman year, both the qualitative analysis and the general chemistry suffered. Therefore a system of identification of simple substances was worked out which did not involve group separations. In one semester, the average student identifies 60 elements, oxides, or salts.

The full-dress qualitative course comes in the second year when those things mentioned above, which the freshman does not master, are stressed. This is a very good introductory course to quantitative and physical chemistry.

All of us seem to find it easier to criticize than to praise; to tear down than to build up; to hinder than to help. The opponents to qualitative analysis have had their say; let us hear from its friends.

S. PORTER MILLER

MONMOUTH COLLEGE
MONMOUTH, ILLINOIS

To the Editor:

I must take exception to one statement in the stimulating article of Dr. Harry F. Lewis on, "The role of the liberal arts college in the professional training of chemists."[1] In comparing the rating of arts and science colleges by the number of graduates achieving the doctorate in chemistry in 1936–45 with a study I made of research published from liberal arts colleges,[2] Dr. Lewis states, "From these we find that only six of the first 14 have published any research papers in the *Journal of the American Chemical Society* in the 23-year period from 1927 to date."

Dr. Lewis' colleges with the 14 highest ratings (Table 3) contain three institutions which are not on my list, not because they failed to produce a publication during the period studied, but because the *College Blue Book* lists them as having professional schools or as granting the Ph.D. degree. These three institutions are Dartmouth, Rice, and Mt. Union.

In seeking to determine the research contributions of a liberal arts faculty it is important that no institution with an engineering, medical, or other professional school be included. And the Ph.D. can hardly be rated as a liberal arts degree.

JOHN R. SAMPEY

FURMAN UNIVERSITY
GREENVILLE, SOUTH CAROLINA

[1] LEWIS, H. F., J. CHEM. EDUC., **28**, 104 (1951).

[2] SAMPEY, J. R., *J. Higher Educ.*, **20**, 208 (1949); *Chem. Eng. News*, **28**, 860 (1950).

Recent Books

INDUSTRIAL SOLVENTS

Ibert Mellan, Industrial Chemist and Consultant. Second Edition. Reinhold Publishing Corporation, New York, 1950. x + 758 pp. 369 figs. 194 tables. 16 × 23.5 cm. $12.

THE second edition of "Industrial Solvents" is a completely rewritten volume expanded to 758 pages from the 480 pages of the first edition [reviewed, J. CHEM. EDUC., 17, 402 (1940)]. It remains essentially a discussion of solvents for lacquers, varnishes, paints, and their individual components. In a separate chapter on the industrial application of solvents, many other uses are pointed out, but the emphasis remains on lacquers.

The first section of the book (112 pages) deals with the general solvent properties in four chapters on the nature of solution, solvents, latent solvents, nonsolvents, vapor pressure, evaporation rate, boiling point, viscosity. The first two of these on theory lean heavily on the side of cellulose esters and ethers used in lacquers. The latter two are fundamental in presenting methods for determining vapor pressures, evaporation rates, and viscosity, graphical methods of presenting these data, tables of azeotropes, and general rules concerning these physical properties.

The following two chapters (122 pages) deal with the industrial application of solvents and safe handling of solvents. Many figures and tables are given to show the uses of solvents in various industries, particularly newer uses. In the safe handling of solvents the flammability, flash and fire points, and explosive limits are stressed. Toxicity is given for a number of groups of solvents. Reference is made to the series of Chemical Safety Data Sheets and one is reproduced to show the detailed safety information available (though no list of components for which sheets are available is given).

The largest section of the book (505 pages) devotes a chapter to each class of solvents, as hydrocarbon solvents, halogenated hydrocarbons, nitroparaffins, amines, alcohols, furfural, ketones, acids, ethers, and esters. Each chapter is replete with tables and graphs of physical properties and solvent ability. The large amount of such data makes this book the first to which an investigator or engineer should turn for physical properties.

Although the stress placed on cellulose derivatives will limit the general usefulness of the book to organic chemists and engineers, the large amount of data of a general nature presented here will make this book one that he will consult frequently to secure solvent ability for new compounds or physical properties for design information.

KENNETH A. KOBE

UNIVERSITY OF TEXAS
AUSTIN, TEXAS

SPECTROCHEMICAL PROCEDURES

Charles E. Harvey, Consulting Spectrographer, Applied Research Laboratories. Applied Research Laboratories, Glendale, California, 1950. x + 389 pp. 75 figs. 9 tables. 16 × 24 cm. $7.

THIS book is the result of Mr. Harvey's long experience in developing new spectrochemical methods and spectrochemical equipment with ARL. It is written both for the practical spectrochemist and for the student. No attempt is made to duplicate the numerous and good theoretical treatments now available in many books. The emphasis throughout is on the practical side. The book abounds in experimental details, many of which have not to the reviewer's knowledge been previously published. The arrangement is logical and easy to follow. Bold face type for paragraph headings facilitates ready reference. The index appears to be adequate. After briefly considering the theoretical basis of spectrochemistry the author takes up in turn the instruments used, photographic calibration, qualitative, semiquantitative, and quantitative analysis.

Of particular value are the chapters on the spectra of the elements and on the variables in a spectrochemical analysis (quantitative). The former discusses, according to periodic groups, the elements most frequently encountered in spectrochemical analyses, giving their sensitivities and likely interfering substances, together with a brief discussion of the applicability of spectrochemical methods for each group. In the chapter on Variables in a Spectrochemical Analysis there is a very thorough treatment of the factors which may affect the reproducibility and accuracy of analyses. The beginning student and experienced spectrographer will both find this chapter helpful.

The final chapter, Setting Up a Laboratory for Operation, will prove valuable for those plant personnel who plan to install spectrochemical methods.

The reviewer feels that this book is a "must" for every spectrochemical laboratory and should be available as a reference for every student who takes a course in the field. The wealth of experimental detail and the reliability of the methods will make it of continuing value as a ready reference. The only flaw we find is that the binding is not sufficiently rugged for a book that will be frequently consulted in daily work.

CONWAY PIERCE

POMONA COLLEGE
CLAREMONT, CALIFORNIA

PLANT VIRUSES AND VIRUS DISEASES

F. C. Bawden, Head, Plant Pathology Department, Rothamsted Experiment Station. Third, revised edition. Chronica Botanica Co., Waltham, Massachusetts, 1950. 335 pp. 5 figs. 54 plates. 59 tables. 18 × 27 cm. $6.

ALTHOUGH organic substances known as viruses were recognized as being associated with both plant and animal diseases in the 1890's their precise role in cell metabolism continues to be elusive. Many forms cause no particular damage to their hosts, but pathogens causing leaf mosaics in tobacco and potato plants as well as leaf curl in sugar beets, to mention only a few, are taking appreciable tolls in agricultural economy. The problems involved have received the attention of many biochemists and pathologists in the last fifteen years. To correlate the information in the extensive literature on the subject has been the task of a distinguished worker in the field, Dr. F. C. Bawden at the Rothamsted Station in England. The third, entirely revised edition of his book witnesses the rapid accumulation of data on plant viruses in even the last eight years.

After dealing with the recognized symptoms of infected plants the author considers the various modes on virus transmission and the nature of insect vectors in particular. In the latter field of study, the work of Black on leaf-hopper transmission of a clover virus, for example, has demonstrated the fact that this virus is maintained for twenty generations through the eggs, after a single, initial infection. The point of biochemical significance in this very recently reported case is that a given virus may play a similar role in the metabolism of both the insect and plant involved.

Virus action may be exceedingly complex, as the chapter on virus strains indicates. A particular virus, for instance, may have quite different effects on different hosts. Furthermore, related strains may produce an inhibitory effect on each other in

the same host while unrelated strains may produce an additive or even intensified effect. It follows that serological analyses of virus complexes, such as are considered in the next two chapters, offer a valuable complement to other techniques of biochemical assay. "The methods have been used to show not only that viruses which were thought previously to be unrelated, because they cause different symptoms or have different host ranges, are related strains, but they have been valuable also in demonstrating that some clinically similar diseases are etiologically different."

From serological considerations the author passes on in the ensuing half of the book to the chemical and physical properties of purified virus preparations, crystallinity, particle size, and types of inactivation. As is well known, the electron microscope has given a greatly increased advantage to the physicochemical approach to the subject, since the particles concerned are under 200 mμ in size.

A concluding chapter on origins points out the relationship of the nucleoprotein extracts, currently known as or associated with viruses, to the many organic compounds, including enzyme systems, involved in cell metabolism. Unique in their property of self-multiplication, viruses may represent, however, metabolites that under certain conditions develop in considerable excess. Certainly, they can no longer be conceived as primordial, protoplasmic units.

Here is a reference book that is invaluable to students and workers in both physical and biochemistry.

GEORGE L. CHURCH

BROWN UNIVERSITY
PROVIDENCE, RHODE ISLAND

● COLLOIDAL DISPERSIONS

Earl K. Fischer, **National Bureau of Standards. John Wiley and Sons, Inc., New York, 1950. vii + 387 pp. 71 figs. 67 tables. 15.5 × 24 cm. $7.50.**

ALTHOUGH the objective of this book is to cover dispersions of solids in liquid media, it is almost entirely devoted to industrial processes involved in producing surface coatings and inks. This limitation eliminates a vast amount of colloid chemical literature of less economic but considerable theoretical interest. It also limits the major attention to particles in the upper range of colloidal dimensions. The dimensional limits of colloidal particles are defined in the book as 1 micron and 1 millimicron, but with the exception of carbon blacks, which range downward to 3 millimicrons, most of the attention is given to particles above 50 millimicrons in diameter. Though this limitation of the book is left for the reader to discover, it is a desirable one in order to retain the book within reasonable dimensions and to focus it upon industrial applications.

The book opens with a very satisfactory discussion on particle size, its meaning and measurement. Here is found a very readable description of the meaning of particle size and the eight different average diameters found in the literature. The measurement of particle size by sieving, optical microscopy, ultramicroscopy, electron microscopy, adsorption of gases and vapors, sedimentation, diffusion, X-ray measurements, and turbidity is given adequate attention. In addition, detailed attention is given to some of the more common pigments.

Following the discussion of particle size are three chapters devoted to the dispersed state and a discussion of such matters as the solid liquid interface, the contact angle, adhesion tension, spreading of liquids on solids, flocculation, and rheological properties. The last-named subject is dealt with in good detail, and plastic flow, thixotropy, and dilatancy have been clearly presented.

The last third of the book is devoted to dispersing methods. It gives a good outline of the influence of surface-active agents and of the current practice with roll mills, ball and pebble mills, disc and cone mills, and dispersion by phase transfer.

Altogether, the reviewer finds this book to be, within the limits of its coverage, a very satisfactory presentation of the subject. Errors have been few and the literature has been covered quite well for the type of audience for which this book was written.

S. S. KISTLER

NORTON COMPANY
WORCESTER, MASSACHUSETTS

● PROGRESS IN COAL SCIENCE

Edited by *D. H. Bangham,*[1] Director of Research Laboratories, British Coal Utilisation Research Association. Interscience Publishers, Inc., New York, 1950. xi + 456 pp. 32 figs. 27 tables. 12 plates. 16 × 25 cm. $7.

THIS book consists of reviews selected from a series of articles which were privately circulated in the Monthly Bulletin of the British Coal Utilisation Research Association during 1943–47. The view was that "a useful purpose would be served by making these available to a wider public. The selection of subjects for this volume . . . has been dictated by the desire to present up-to-date information in fields inadequately covered by existing textbooks. It is hoped that this choice will be such as to appeal to a wide range of readers: to students . . .; to scientists in industry . . .; and . . . to workers in all countries engaged in the task of advancing our knowledge of coal and of its technology and utilisation."

The book covers five main fields of discussion which in turn are subdivided into 26 chapters.

In the first section entitled "Modern Experimental Techniques" are contained the following chapters: Crystallographic Techniques in Chemical Analysis, Applications of Absorption Spectroscopy to Organic Compounds, Mass Spectrometry for Chemical Analysis, Chromatographic Adsorption Analysis, Photographic Techniques in Combustion Research, and Measurement of Flame Emissivity.

The section on "Fine Particles" deals with Fine Powders and Particles in Industry, Particle Size Measurement, Assessment of Specific Surface by Adsorption Methods, Non-Mechanical Methods of Size Reduction, and Grindability of Coal.

Chapters on Formation of Humus and its Relation to Coal, Analysis and Sampling of Solid Fuel, Occurrence of Rare Elements in Coal Ashes, and Coal Petrology are found in the section dealing with "Coal Constituents."

Under the "Organic Chemistry of Coal Products" are chapters on: Action of Chemical Reagents on Bituminous Coal, Action of Oxygen on Coal at Moderate Temperatures, Progress of Coal Tar Research in the Period 1910 to 1940, Recent Work on Coal Tar, and Hydrocarbon Synthesis.

The last section deals with "Chemical Aspects of Combustion and Gasification" and includes the following chapters: Combustion in Fuel Beds, Some Kinetic Aspects of Gas Phase Combustion, Sulfur Compounds in Combustion, Effect of Inorganic Compounds on the Behavior of Coals and Cokes, Coal Ash: Chemical Composition as a Guide to Behavior in Furances, and Recent Developments in Gasification.

The purpose of the book in making more generally available to coal investigators the recent advances in specialized fields has been fulfilled. However, because of the time lag involved, this purpose would be better served in the future if the original BCURA articles themselves were given wider distribution.

The chapter dealing with non-mechanical methods of size reduction is, so far as the reviewer knows, the only published comparison of the effectiveness of the various types of equipment dependent on impact, attrition or explosion effects on the particles. It is stated that "these novel methods of pulverizing are, in general, less satisfactory than conventional mechanical methods. For superfine grinding mechanical mills can hardly compete with the attrition type mill and the requirements of the gas turbine lend impetus to research on non-mechanical mills of all types." In the discussion on "grindability of coals" it is emphasized that different types of grinding tests place coals in different orders of

[1] Deceased.

"grindability," and that the factors responsible for this anomaly are as yet imperfectly understood.

The chapter on humus and its relation to coal formation properly emphasizes the great confusion which exists in this field. The statement "evidence is still lacking as to whether the products of degradation of cellulose could, under certain conditions, acquire an aromatic structure" should be qualified to refer to natural conditions. Such cyclization under laboratory conditions has been well established.

In the discussion of analytical methods the following statement is of interest: "The 'agglutinating value' or coking index test is slowly passing from favor in British laboratories. The time is ripe for the development . . . of a test on a new basis."

The question of the rare element content of coal ashes is of general interest and the author of the chapter on this subject is the late V. M. Goldschmidt, an authority in this field. A table giving the amounts of rare elements in various coal ashes is shown, together with a comparison with the average amount of the same element found in the earth's surface. Enrichment factors as high as 1600 to 1 are reported.

In the chapter on petrology, C. A. Seyler reviews the discovery of the value of the oil immersion lens for optical examination of coal surfaces and presents evidence for the view that the coalification process proceeds in discreet steps. The optical components which have different and measurable reflectances are given numbers. It is said that "the recent discovery that fusain and charcoal consist of the same optical components No. 6 to 9 . . . appears almost conclusive evidence of some form of the forest fire theory."

The statement in the chapter dealing with hydrogenation that "Bergius found that 'cracked' coal molecules could be hydrogenated" is somewhat misleading since there is definite evidence that coals can be degraded to soluble products by hydrogenation at temperatures well below those at which thermal decomposition appears.

The chapter on coal tar research between 1910 and 1940 is a translation of an article by Otto Kruber which appeared in *Zeitschrift für angewandte Chemie* in 1940. The interesting statement is made that "even in (Germany) the science and technology of coal tar very often have little in common. Thus, even tar experts are sometimes ignorant of the composition of coal tar."

The final chapter on recent developments in gasification is by D. T. A. Townend, Director General of BCURA, and describes briefly the science and technology of gasification. Lurgi, Winkler, Imperial Chemical Industries, Koppers, and Thyssen-Galoczy units are pictured and discussed.

The printing and format of this book are excellent and very few typographical errors were noted: on p. 299 the 4-methyl indole is shown with an OH_3 group; on page 302 the structural formula for 2,6 dimethyl naphthalene is incorrect, and on page 328, line 7, the word "tars" should be substituted for "tests."

H. C. HOWARD

Carnegie Institute of Technology
Pittsburgh, Pennsylvania

CAROTENOIDS

Paul Karrer **and** ***Ernst Jucker,*** **Director and Research Assistant, respectively, at the Chemical Institute of the University of Zurich. Translated and revised by** ***Ernest A. Braude,*** **Lecturer in Organic Chemistry, Imperial College of Science and Technology, London. Elsevier Publishing Co., Inc., New York, 1950. x + 384 pp. 31 figs. 56 tables. 2 plates. 17 × 25.5 cm. $8.50.**

This 384-page revised translation of the Swiss edition (1948) deals primarily and in a thorough manner with the physical and chemical properties of the increasingly important natural pigments of the carotenoid class.

The volume is divided into two chief sections: (1) a general part, with nine chapters, comprising nearly the first third of the text, and dealing with such subjects as occurrence, detection, identification and isolation of carotenoids, their chemical, physical, and isomeric properties, synthesis and distribution in nature; (2) the longer special part, comprising five substantial chapters, takes up the carotenoid biochromes group by group: the hydrocarbons, the hydroxyl members, those of known structure carrying one or more carbonyl groups, carotenoid carboxylic acids, and finally an extensive list of carotenoid compounds whose structures yet remain partly or competely unknown.

Organic chemists and biochemists will find this volume to be a valuable reference, in that the structural formulas of most of the known carotenoids are shown (Chapter IV); generous series of tables are presented, *e. g.*, on the relative position of carotenoids on chromatograms, with correlative information as to number of hydroxyl, carbonyl, and ether groups, conjugated and isolated ethylenic double bonds (Chapter III); and in Chapter VII, a similar list of the absorption spectra (in CS_2) of natural carotenoids, the formula of each, and its number of double bonds, hydroxyl, and carbonyl groups.

In the special section information concerning each individual compound is given as thoroughly as current knowledge of the subject has made possible. Thus the reader obtains information on the history, occurrence, natural sources, preparation, chemical constitution, possible synthesis, diagnostic chemical and physical properties, synthetic derivatives, and the absorption spectra of all such derivatives in several solvents. Physiological properties are mentioned where known. Some of the sections on individual carotenoids are extensive and thorough, while others remain necessarily brief, awaiting results of further study.

At the end of the text there appear two colored plates illustrating the crystalline habit of 12 of the commoner carotenoids as obtained from named solvents. Following these is a series of curves depicting the light-absorption spectra of various carotenoids in given solvents. These graphs are taken from various publications, and therefore vary somewhat in length and subdivisions of coordinates, and in units of the ordinate.

In view of the value and thoroughness of the chemical parts of this book, readers seeking detailed biochemical or physiological information are apt to meet with some disappointment, and biologists, seeking chemical information, may become prejudiced to some extent, because of the presence of certain incomplete or inaccurate statements in the biochemical and physiological realm of the subject, as well as occasional departures from accepted biological nomenclature and from conventional classification of organisms. The reviewer appreciates the great task which the writers have essayed, and the successful achievement of the purely chemical portion of that task. But it is impossible not to regret the omission of many corrections which could have been made in the manuscript stage by one or two botanical and zoological referees.

On page 6 we read "...part of the pigments is excreted unchanged, while the remainder is absorbed." Actually, it has been known for a number of years that some carotenoids may be neither absorbed by the animal nor excreted unchanged, but chemically altered to colorless compounds in the alimentary tracts of certain animals, notably in the ruminants.

On page 82, the following statement is found: "It is not known with certainty whether the pigments are synthesized by the (invertebrate) animal or contained in the food." No case has been established of an animal's ability to synthesize carotenoids *de novo*, whereas it is known that these pigments are available in the food which the animals utilize. Numerous invertebrate animals, like certain of the vertebrates, are furthermore highly selective in the type of carotenoid absorbed from the detrital, plant, or animal food taken in. Few references seem to have been made to such facts, and numerous important publications relative to carotenoid metabolism appear to have been surveyed only in part, or completely overlooked.

There is, unfortunately, no index of authors, and the general subject index is limited to 7 pages of terms or headings of a chemical nature. There is, however, a 15-page index of plant and animal sources of carotenoids.

DENIS L. FOX

Scripps Institution of Oceanography
La Jolla, California

● COLLISION PROCESSES IN GASES

F. L. Arnot, late Lecturer in Physics, University of Sidney. Fourth edition. John Wiley & Sons, Inc., New York, 1950. viii + 104 pp. 37 figs. 11 × 17 cm. $1.25.

● THE KINETIC THEORY OF GASES

Martin Knudsen, Professor of Physics, University of Copenhagen. Third edition. John Wiley & Sons, Inc., New York, 1950. vii + 64 pp. 20 figs. 11 × 17 cm. $1.25.

● X-RAYS

B. L. Worsnop and *F. C. Chalklin.* Third edition. John Wiley & Sons, Inc., New York, 1950. viii + 126 pp. 47 figs. 11 × 17 cm. $1.25.

● ATOMIC SPECTRA

R. C. Johnson, Master of Queen's College, University of Melbourne, Melbourne, Australia. Second edition. John Wiley & Sons, Inc., New York, 1950. viii + 120 pp. 38 figs. 11 × 17 cm. $1.25.

THESE four little books are from the series known as "Methuen's Monographs on Physical Subjects," published in England by Methuen and Co. Ltd., of London. Each one is an exposition of a restricted field of scientific inquiry, written for readers of "average scientific attainment." Mr. Arnot's monograph is restricted to collision processes in gases at low pressures where the mean free paths of the colliding particles are large compared to their diameter. Under these conditions single collisions may be studied. Such collision processes as glow and arc discharges, or diffusion, which occur at higher pressures and must be studied by statistical methods, are excluded from the book. Professor Knudsen writes principally of his own investigations on the effusion of gases through holes or tubes, heat conduction, coefficient of accommodation, etc., and deals only with that part of the kinetic theory that he has found useful in his own researches. Such topics as Maxwell's law for the distribution of velocities of gas molecules, or equations of state for nonideal gases are therefore omitted. The monograph on X-rays has sections on the production of X-rays, X-ray spectrometry, Bohr's theory of emission spectra, the continuous X-ray spectrum, the photoelectric effect of X-rays, and on the absorption, scattering, diffraction, reflection, and refraction of X-rays. Mr. Johnson discusses atomic spectra, using "the picturesque language of the older quantum theory, talking freely about such things as electronic position and orbital motion," for he maintains that "the experimental physicist and chemist inevitably cling to this wreckage lest they be drowned in the mathematical sea." He nevertheless indicates those points at which the new quantum theory or wave mechanics are particularly illuminating. Each author includes a bibliography or suggestions for detailed reading so that those whose interest is stimulated may consult other sources for a more comprehensive treatment of the subject.

The advertising statement on the jacket that the series is intended to supply "a compact statement of the modern position in each subject" should mislead no one. "Modern" means "not ancient," and is not to be confused with contemporary. The first two of these books were written in 1933 and have been little changed in succeeding editions; such phrases as: "a paper by Penney has just appeared" [*i.e.*, in 1932], or "a very recent investigation" [also in 1932] might well have been changed in the second, third, or fourth editions. "X-rays," although first published in 1930 under the authorship of Worsnop, was revised by Chalklin in 1946, and "Atomic Spectra" was first published in 1946.

However, neither the failure to revise some of these monographs nor their necessarily circumscribed nature detract much from their value. They are not treatises, but more akin to an intimate explanation by the author of his own research interests. The student about to undertake research can learn from these books how one research problem leads to another, how the work of a single investigator fits in with the discoveries of others in the same field. For the experienced research worker these books can supply knowledge of scientific inquiry outside one's own field of research, and perhaps indirectly serve as a stimulus to one's own investigations. The pocket size of these monographs makes them very convenient for reading in odd moments, such as on the train or while waiting for one's wife to dress or for the fish to bite. Their modest price, and facile and authoritative style make them very attractive.

WALTER B. KEIGHTON

SWARTHMORE COLLEGE
SWARTHMORE, PENNSYLVANIA

● PHOTONS AND ELECTRONS

K. H. Spring. John Wiley & Sons, Inc., New York, 1950. vii + 108 pp. 38 figs. 11 × 17 cm. $1.75.

THIS book, one of "Methuen's Monographs on Physical Subjects," is a worthy newcomer to that excellent series.

The object of the book is to present a fairly brief account of present-day knowledge concerning the more important ways in which electrons interact with radiation. The phenomena dealt with are not primarily those involving low energies (of the order of one or a few electron volts), in which the chemist is mainly interested, but rather those involving high energies (kev. and mev.), of interest more to the radiation physicist. The book is written for readers of average scientific attainments, and the treatments given presuppose a general familiarity with quantum theory and elementary radiation theory; there is, however, a chapter on "Fundamentals" in which the salient points of the presupposed theoretical background are reviewed very briefly. For derivations of the many equations given the reader is for the most part referred elsewhere.

Processes in which photons lose energy to electrons, namely the photoelectric effect and the Compton effect, are first treated, and then the reciprocal processes, in which electrons lose energy with the production of photons, as in Bremsstrahlung and Cerenkov radiation, are discussed. After a brief introduction to the nature of the positive electron, the phenomenon of pair-production and the reciprocal process of electron-positron annihilation are treated. In the final chapter cosmic ray phenomena are described and treated in the light of the processes mentioned earlier, and the theory of shower development is summarized. Throughout the book considerable emphasis is placed on the directional aspects involved in the processes treated. In many places in the book experimental results are presented and correspondence with theory is discussed.

DAVID P. SHOEMAKER

CALIFORNIA INSTITUTE OF TECHNOLOGY
PASADENA, CALIFORNIA

● A TEXTBOOK OF GENERAL ORGANIC CHEMISTRY: STEREOCHEMISTRY

E. de Barry Barnett, formerly Head of the Chemistry Department, Sir John Cass Technical Institute. Sir Isaac Pitman and Sons, Ltd., London, 1950. ix + 169 pp. 14 × 22.5 cm. $4.

IT SEEMS unfortunate to this reviewer that the author has published this material as a separate book instead of following his original plan of using it as a chapter in an advanced textbook of organic chemistry. He says in the preface that "no textbook on stereochemistry has appeared in English since the publication of the second and final edition of Stewart's 'Stereochemistry' in 1919." Although this statement technically is correct, an objection to it can be raised, since the chapter entitled "Stereoisomerism" by Shriner, Adams, and Marvel in Gilman's "Organic Chemistry," published first in 1938 and revised in 1943, is a more extensive treatment than that of the book under review. Moreover, in the reviewer's opinion, this book is less clearly written

and, except for a few instances, is no more up-to-date than the chapter by Shriner, Adams, and Marvel.

A few examples will indicate the nature of the objections that can be made to the book. Since reference is made to the theoretical work of Boys, of Condon, and of Eyring, one would expect reference also to that of Werner Kuhn and of Kirkwood. Similarly it is difficult to understand the failure to mention the experimental work of Werner Kuhn, who first demonstrated the formation of an optically active compound by the action of circularly-polarized light, and of Prelog and Wieland, who resolved Troeger's base and thus prepared the first optically active compound, the activity of which is due to tricovalent nitrogen atoms. Contrary to the author's statement, it has been shown that the direction of approach of a group during a Walden inversion is not governed by the electrical properties of the groups taking part in the displacement reaction. The author writes formulas for the conjugate acids of esters and of carboxylic acids with the protons on the alkoxyl or hydroxyl oxygen atoms, whereas the currently preferred location is on the carbonyl oxygen. An appendix of thirteen pages is devoted to the construction of models composed of solid tetrahedra. Such models are hardly the most helpful type for visualizing the configurations of molecules.

This reviewer hesitates to find fault with minor matters that have no bearing on the scientific aspects of the treatment. However the rather frequent typographical errors, the misspelling of authors' names, the inconsistencies in the hyphenation and compounding of words, and the erratic inclusion of authors' names in references at the ends of chapters cannot help but annoy the reader.

CARL R. NOLLER

STANFORD UNIVERSITY
STANFORD, CALIFORNIA

● ORGANIC CHEMISTRY

P. J. Durrant, **Selwyn College, Cambridge University. Longmans, Green and Co., Ltd., New York, 1950. xi + 544 pp. 60 figs. 18 tables. 14.5 × 22.5 cm. $3.75.**

THIS book offers an interesting contrast in approach to its subject in comparison with American elementary textbooks. Theory and laboratory practice are treated concurrently, the text being divided about equally between the two. Durrant's text is a little more detailed than most one semester American books, but by judicious selection of material could well be used for such a course. Since Durrant has laboratory procedures for each class of organic molecules interspersed throughout the book, it should be used as both laboratory manual and lecture text.

In Chapter 1 the electronic structure of the elements is considered in more detail than in most elementary American texts. The last part of the chapter is devoted to a discussion of resonance using CO_2 as a model compound.

Chapter 2 is called The Scope of Organic Chemistry. In this section of 23 pages the nomenclature and type formulas for the important classes of organic compounds are quickly surveyed. Aliphatic as well as aromatic are considered. Also a hasty explanation of optical and geometrical isomerism is given. This chapter was a disappointment since the title leads one to believe that the over-all objectives of organic chemistry were to be discussed rather than merely an outline of nomenclature.

Chapter 3 on Laboratory Technique contains a large number of good drawings of common types of organic laboratory equipment. Good discussion of distillation, crystallization, melting points, etc., is given. Chapter 4 describes the procedures used for determination of the molecular constitution of organic substances. Qualitative and quantitative determination of the elements C, H, O, S, and halogens are explained. The discussion is quite thorough and well illustrated with drawings of the necessary apparatus.

With Chapter 5, page 104, Durrant continues, where many American books begin, with a study of paraffins. Aliphatic chemistry is covered in the next 198 pages. Then follows a chapter on optical and geometrical isomerism, one on polyhydric alcohols, and then one on carbohydrates. The next 122 pages are given to aromatic chemistry. Chapter 29, 34 pages, is a recapitulation of all the important organic reactions in tabular form with a brief summary statement of the usefulness and limitations of each. This chapter should be welcomed by students preparing for exams. The last chapter in the book discusses how organic compounds are prepared industrially from the four fundamentals, carbohydrates, oils and fats, coal, and petroleum. Students will find this a most interesting chapter. Durrant emphasizes a point often neglected: that the various countries frequently use quite different raw materials to make the same chemical.

There are no questions at the end of the chapters, but at the end of the book a section of 314 review questions is included.

CORWIN HANSCH

POMONA COLLEGE
CLAREMONT, CALIFORNIA

● CHEMICAL ENGINEERING COST ESTIMATION

Robert S. Aries **and** ***Robert D. Newton,*** **Adjunct Professor of Chemical Engineering, Polytechnic Institute of Brooklyn, and Chemical Engineer, Charles Pfizer and Co., Inc., respectively. Chemonomics, Inc., New York, 1950. 109 pp. 27 tables. 67 graphs. 21.5 × 28 cm. $5.**

ONE important function of the chemical engineer is to estimate the cost of the new plant which will produce the chemist's brain child. If the cost is too high the completed research project remains only that. This book gives charts and rules based on published cost figures for installed equipment which will permit an engineer to estimate with a fair degree of accuracy the cost of the required plant. The material presented here is largely a collection of graphs and tables previously published in *Chemical Engineering* and elsewhere. Unfortunately, the literature citations are frequently incomplete, and time is wasted seeking the original article. Because this is one of the few collections of such information the book will be consulted frequently by engineers making cost estimations.

KENNETH A. KOBE

UNIVERSITY OF TEXAS
AUSTIN, TEXAS

● HANDBOOK OF ANTIBIOTICS

A. L. Baron, **S. B. Penick & Co., New York. Reinhold Publishing Corp., New York, 1950. viii + 303 pp. 16 × 23.5 cm. $6.50.**

"HANDBOOK of Antibiotics" is a descriptive manual condensed from the accumulated literature existing in late 1949 and, in some instances, early 1950. It presents abbreviated details of history, production, chemistry, bacteriology, and pharmacology for many of the 141 antibiotics listed. The author has considered an antibiotic to be a metabolic product that is antagonistic in low concentration to the growth of one or more microbial species. Origin is not restricted to products of microbial metabolism; thus, such materials as plumbagin and tomatine are included.

Discussion of each antibiotic includes portions of the following details: history—discoverer, productive organism, synonyms; production—type of fermentation, formulation of media, expected yields, methods of isolation and purification; chemistry—constitution, properties, reactions, derivatives; bacteriology—method of assay, microbial spectra *in vitro* and *in vivo*, mode of action, factors affecting activity, microbial resistence; pharmacology—toxicity, physiology, blood level, spinal fluid level, fetal blood level, absorption, excretion, diffusion. The attempt to include all antibiotics has made the manual erratic in balance. Many of the entries are discussed thoroughly; others, however, receive little attention. Although this situation results unquestionably from the scarcity of publications relative to certain antibiotics,

such limitations challenge the title's implication—the compilation could be described as a handbook of selected antibiotics. Present-day emphasis upon antibiotic research will make frequent revision of the work desirable.

The book is admirably objective in presentation and, in spite of its enforced disproportions, should be a time saver for those who have been obliged to search the literature for technological facts. Its usefulness is increased somewhat by the inclusion of two supplementary chapters. In one of these, Henry Welch discusses briefly the needs for and methods of certification of antibiotics by the U. S. Food and Drug Administration. In the other, W. J. Derenberg clarifies the means of selection and protection of commercial names applied to both basic and secondary antibiotic products. He lists eighty antibiotic trade-marks that were registered or about to be registered at the U. S. Patent Office as of May, 1950. Products listed are those that contained a currently available basic antibiotic (penicillin, streptomycin, chloramphenicol, aureomycin, bacitracin, tyrothricin, terramycin).

More than 900 titled references are given (the bibliographies of penicillin and streptomycin are restricted to books). Appended are a short glossary of terms, a table of antibiotic-producing organisms and their products, an author index, and an index of organisms.

WILLIAM D. ROSENFELD

California Research Corporation
La Habra, California

● PLASMA PROTEINS

Volume II of "Symposia on Nutrition of the Robert Gould Research Foundation." Edited by *John B. Youmans*, Dean and Professor of Medicine, School of Medicine, Vanderbilt University. Charles C Thomas, Publisher, Springfield, Illinois, 1950. xviii + 352 pp. 85 figs. 49 tables. 15.5 × 23.5 cm. $6.50.

This volume consists of papers presented at a Symposium on Proteins held on September 23–24, 1949, under the auspices of the University of Illinois, College of Medicine. A wide range of interests is represented, going beyond purely nutritional problems involving the plasma proteins, and including a discussion of the "Fractionation and properties of the proteins of normal human plasma" by J. L. Oncley and of "Equilibrium treatments of the binding of ions to proteins illustrated by serum albumin, chloride, and thiocyanate" by S. Howard Armstrong, Jr.

More closely related to the nutritional aspects are papers on "Some aspects of the biosynthesis of amino acids" by David Shemin; the "Use of C^{14}-labeled amino acids in the study of peptide bond synthesis" by Ivan D. Frantz, Jr., and Paul C. Zamecnik; and the "Physiological properties of amino acid antagonists" by Karl Dittmer.

Various aspects of the problem of plasma protein formation are treated by S. C. Madden in "Plasma protein in disease states"; by Irving M. London in "Studies on the rates of formation of serum proteins in man"; by Bacon F. Chow in a discussion of "The role of dietary proteins on plasma protein regeneration"; by James B. Allison in "Some relationships between diet, protein stores, and plasma proteins"; by Robert Elman in "Clinical problems in hypoproteinemia due to protein deprivation"; and by Abraham White and Sidney Roberts in considering the "Influence of the adrenal cortex on plasma protein formation and utilization."

"Studies on the fate of intravenously administered human plasma proteins in idiopathic hypoproteinemia and in osteoporosis" are presented by Fuller Albright, Anne P. Forbes, Frederick C. Baxter, Edward C. Reifenstein, Jr., Dorothy Bryant, Lowell D. Cox, and Eleanor F. Dempsey. The problem of "The metabolism of human serum albumin administered to man" is also considered by Richard D. Eckhardt and Charles S. Davidson.

Problems of edema in protein deprivation are dealt with by Hugues Gounelle in a paper entitled "Studies concerning nutritional edema and proteins" and by D. Mark Hegsted, Norman Zamchek, Cheng-Fa Wang, and Melvin B. Black in "Studies on protein deficiency and temperature in relation to edema."

"Studies on liver proteins," by Philip P. Cohen and S. Sorof; "Protein metabolism in relation to resistance and to infection," by Paul R. Cannon; and "Quantitative immunochemical studies of antibody-containing serum fractions obtained by salt precipitation, alcohol precipitation, or delipidation," by Michael Heidelberger and Robert C. Krueger complete the subjects presented.

From its nature, this volume does not, as the title "Plasma Proteins" might imply, present a comprehensive treatment of the plasma proteins. However, the aspects of particular interest in relation to nutrition are quite well covered. Indeed, it is difficult to imagine that as much material could have been presented in the two-day session as is published, without completely exhausting the participants. I would suppose, for example, that the oral presentation of the problem of binding of ions to proteins was less complete from the mathematical standpoint. This scholarly paper is of interest not only because of its content, but because it may strike some readers as rather remarkable that it was written by a Professor of Medicine.

Taking the material in a somewhat different order than that in which it has been arranged for publication, the subject of the symposium may be developed with considerable logic. The evidence regarding the carbon sources of the nonessential amino acids is reviewed, together with the role of amination and transamination in their biosynthesis. The next step leads to the discussion of the incorporation of amino acids into proteins in a single surviving tissue such as liver, hepatoma, or leucocytes, or into plasma proteins produced *in vitro*.

Plasma protein formation *in vivo* is considered from the point of view of the rate of synthesis in the normal individual, with respect to the influence of disease on plasma protein formation, and as influenced by diet. The possibility of factors other than the amino acid composition having an influence on the effectiveness of proteins in making plasma protein regeneration possible is discussed by Dr. Chow, and both he and Dr. Allison emphasize the fact that the nature of the protein provided determines whether it will be most effective in the formation of plasma protein or some other tissue protein. This does not mean, however, that one tissue may not be used to contribute to the effective formation of another, and the work of both Albright and coworkers and of Eckhardt and Davidson shows clearly that a considerable portion of injected serum albumin is effectively used for the formation of some other tissue proteins, and that its metabolic effect is significantly different from that of ingested serum albumin.

Practical considerations in the treatment of hypoproteinemia are dealt with at some length, and the problem of edema is presented both from the point of view of experience with nutritional edema in France during the war and from an experimental standpoint. Both the importance of protein and the operation of other factors are stressed.

A comparison of this symposium with, for example, the symposium of the New York Academy of Sciences on Proteins and Protein Hydrolysates in Nutrition, makes it clear that much progress has been made during the last five years. Although the method of presentation in the present volume varies greatly with the particular author, the presentation is uniformly competent. A good deal of the material has been presented in other reviews, and though the symposium must have been of great interest to the participant, its publication represents some duplication of other available material. This is particularly evident in the case of the article on "Fractionation and properties of the proteins of normal human plasma." Dr. Oncley was faced with a very difficult problem in reviewing a subject which has been reviewed several times previously. Despite the fact that the review was well done this unavoidably detracts from its interest. This is an unhappy type of comment to be forced to make, but one is tempted to speculate on whether it will become necessary to publish a review of reviews, symposia, and monographs.

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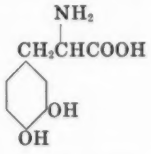

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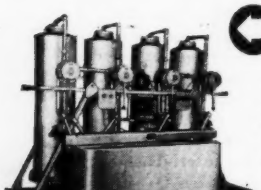

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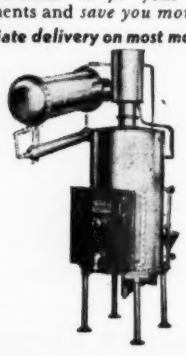

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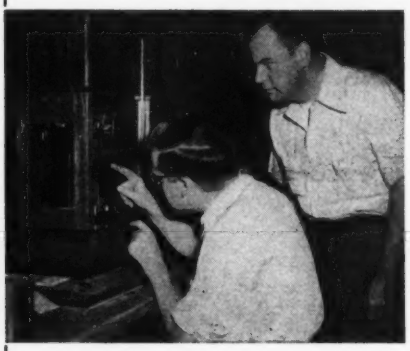

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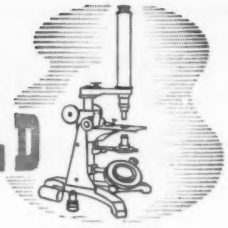

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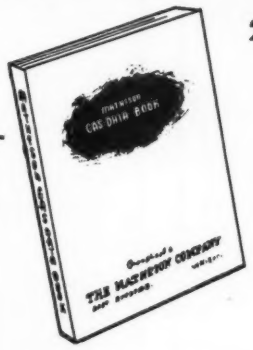

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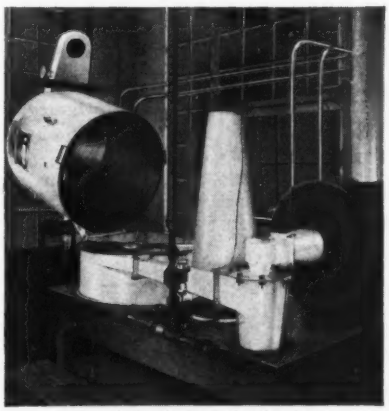

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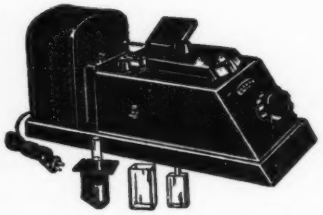

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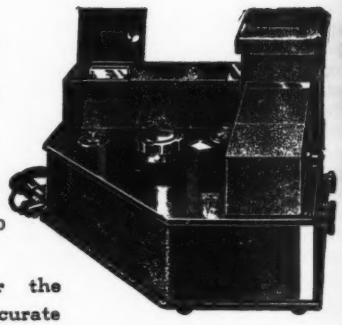

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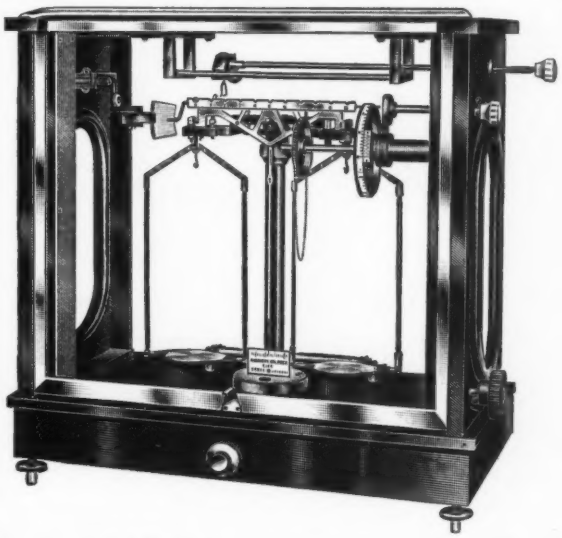

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Out of the Editor's Basket

Hand Torch

A simple hand torch permitting adaptation to all gases at the discretion of the operator has recently been introduced by Hoke, Inc., Englewood, New Jersey. It is the Hoke Jewel Torch, Multi-Use Unit. It eliminates the necessity of acquiring additional torches for infrequent or specialized requirements.

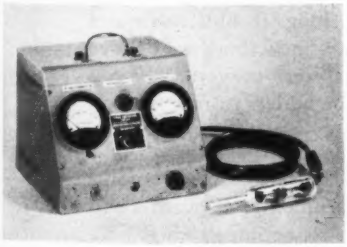
This new torch can be safely used for working pyrex glass and quartz as well as for many other routine operations such as silver soldering, lead burning, ampoule sealing, etc. Designed with "one hand-finger tip control," its flames are instantly adjustable as to size and intensity.

A striking feature of this torch is the ready accessibility of the mixing chamber components. Removal of a single hex nut permits the interchange of injectors and mixing discs. The injector need be changed only when oxygen is being replaced by air.

Constructed from bar and forged brass the torch is attractively finished in nickel.

New Inexpensive Leak Detector

Operating on the principle that the tendency of hot platinum to emit positive ions is enormously stimulated by infinitesimal traces of halogens or their compounds, a new and extremely sensitive inexpensive leak detector for use in vacuum systems down to 1 micron Hg has just been announced by Distillation Products Industries, Division of Eastman Kodak Company.



The new detector, known as the Leak Detector, Model LD-01, is expected to particularly appeal to those who have only occasional need for a leak detector and whose purpose is merely to establish the presence of a leak too small to be detected by more obvious signs.

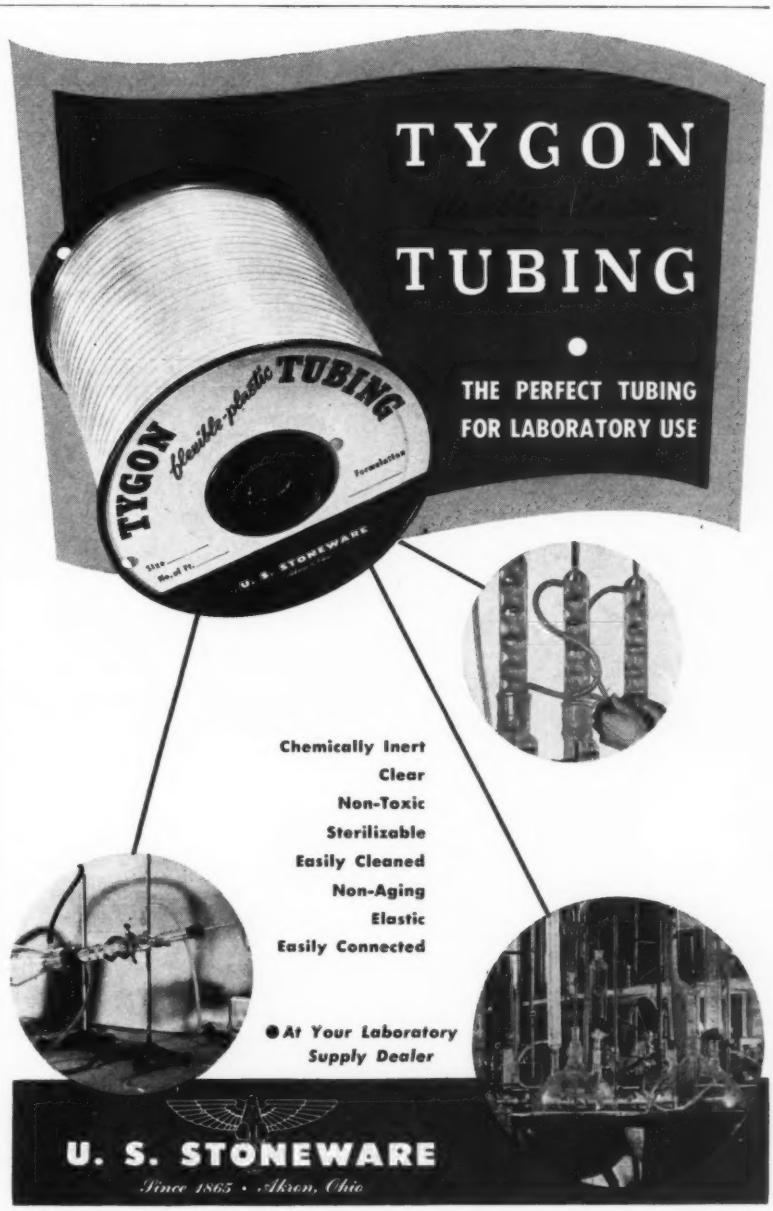
In operation the glass sensitive tube of the leak detector is sealed into the system under test and the system is then pumped down to a moderate vacuum. A jet of harmless Freon gas, readily available in small cylinders (or other halogen-containing gas), is then directed against portions of the system suspected of leaking. When the jet strikes a leak there is a sudden deflection of the meter needle on the instrument face, because the gas—entering the system—instantly increases the ability of a hot platinum anode in the sensitive tube to emit positive ions.

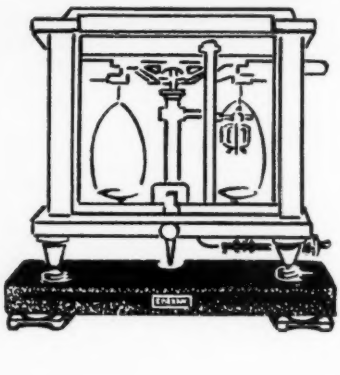
The Model LD-01 circuit is a simple one, employing only three standard radio tubes and housed in a cabinet $10^1/_4$ in. wide, $7^1/_2$ in. high, and 8 in. deep. This cabinet comes on a dolly for convenience in working around large systems. The sensitive tube is 5 in. long, $1^5/_{16}$ in. in diameter, and has a 3 in. tubulation, $^7/_{16}$ in. outside diameter. This may be connected to a system under test with a fitting of the stuffing box type.

Further information on the detector and its use may be obtained directly from Distillation Products Industries, Rochester, New York.

Quartz Monochromator

Those who require a source of monochromatic light for microscopy or other purposes can now secure it in a very simple device which utilizes the rotary power of quartz. The new device not only





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This non-magnetic base is designed for supporting delicate mechanical, optical or electrical instruments. It absorbs frequencies as low as 10 cycles per second; for optimum vibration damping load should be 20 to 40 pounds. This non-ferrous reinforced concrete platform with black wrinkle finish is mounted on 4 Lord multiple shock absorbers. It weighs 50 pounds and measures 22″ by 12″ by 3¼″ high. A threaded insert in rear center of the platform holds a support rod. No. 78-902 Instru-Mount is $32.50. No. 78-903 aluminum support rod 18″ long is $1.25.

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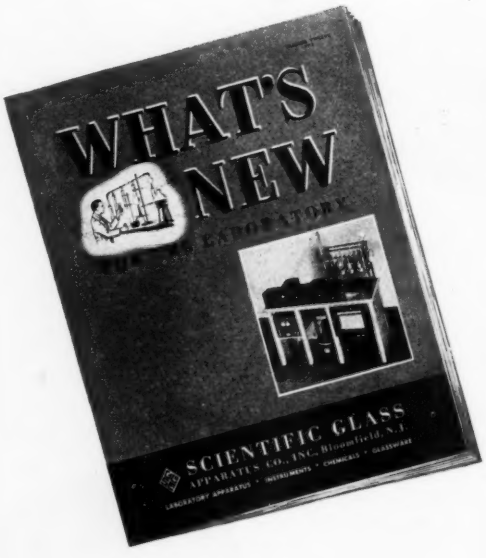

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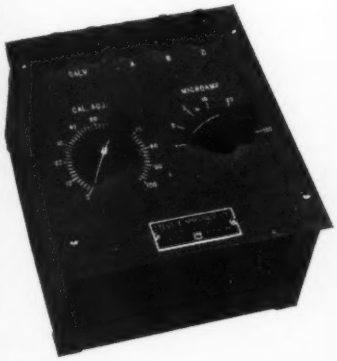
In the quartz monochromator, white light from a tungsten source is passed through basal sections of optical quartz laminated to polaroid sheets. By a synchronous rotation of the quartz sections, the wave length of transmitted light can be varied continuously through the visible spectrum. An average band width of 150 Å. is produced. An adjustable support is provided for tilting the monochromator in line with light source and the miscroscope or other apparatus used.

Inquiries should be addressed to: Cambridge Thermionic Corporation, Concord Ave., Cambridge 38, Massachusetts.

Calibrating Unit

A new Students' Calibrating Unit enables schools to supplement physical chemistry courses with laboratory experiments in polarography without investing in expensive equipment or rigging up makeshift apparatus. By adding this Calibrating Unit to a circuit containing a standard lab potentiometer, galvanometer, and electrodes, students can make their own polarographic measurements.



Polarizing voltage is supplied by the user's own potentiometer. The new Unit, a modified Ayrton shunt, calibrates the user's galvanometer so that it provides direct readings of diffusion current passing through the test solution. By causing thermals in the galvanometer to oppose, the Unit minimizes drift. Since it keeps critical damping resistance across the galvanometer terminals, it helps the galvanometer follow current changes quickly and accurately. With a truly linear galvanometer, readings show less than 1 per cent error.

The wide choice of ranges—1, 2, 5, 10, 20, 50, and 100 microamperes—adapts the Unit to a broad variety of polarographic analyses.

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Ultramax Buret

A greatly improved model of the Ultramax Buret has recently been introduced by the Emil Greiner Co., 20–26 North Moore St., New York 13, New York.

In the new instrument the operation of the teflon plug has been changed to give positive turning and independent adjustment of pressure. The round knob has been replaced with a hexagonal shape to prevent rolling of the buret on a horizontal surface. This knob and lock nut are now made of acid- and alkali-resistant Vinylite instead of catalin, as previously employed. New methods of machining the plug have eliminated any possibility of leakage.

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"Playground of a Scientist"

On the occasion of the retirement in 1949 of Eva V. Armstrong as Curator of the Edgar F. Smith Collection in the History of Chemistry at the University of Pennsylvania, her friends issued in her honor a reprint of the article "Playground of a Scientist" which she had originally published in 1936. In this she traced the life of Edgar Fahs Smith and the growth of his interest in the history of chemistry in terms of some of the volumes in his library. The little reprint, available from the Smith Collection, is a delightful addition to the literature of the human side of chemists and chemistry

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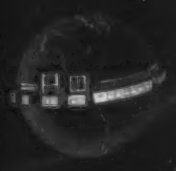

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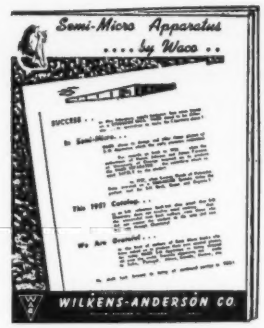

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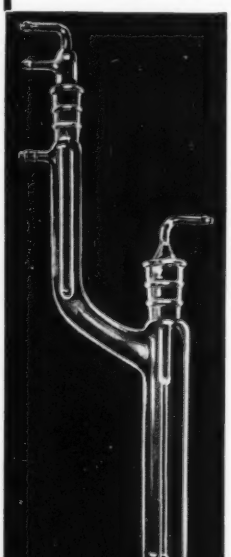

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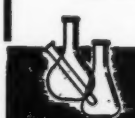

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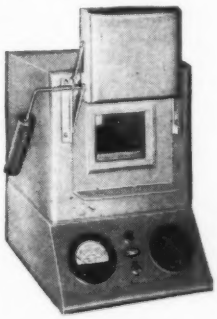

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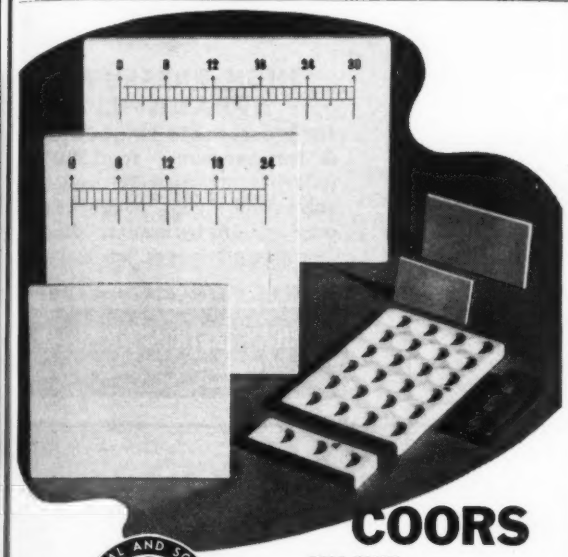

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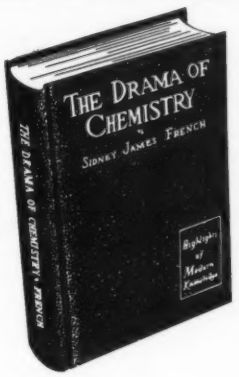

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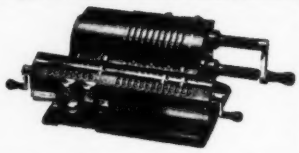

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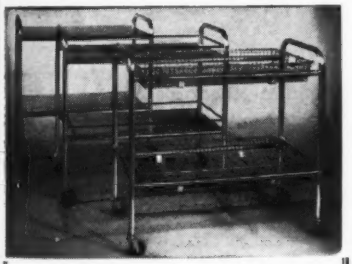

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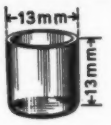

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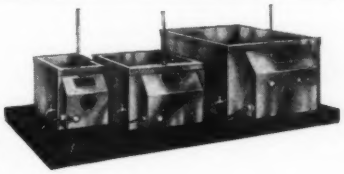

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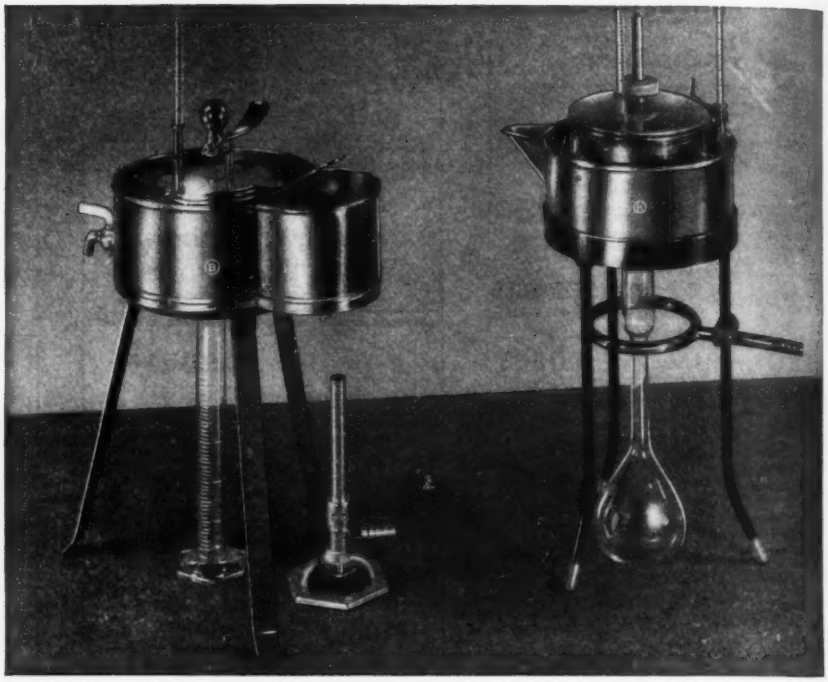

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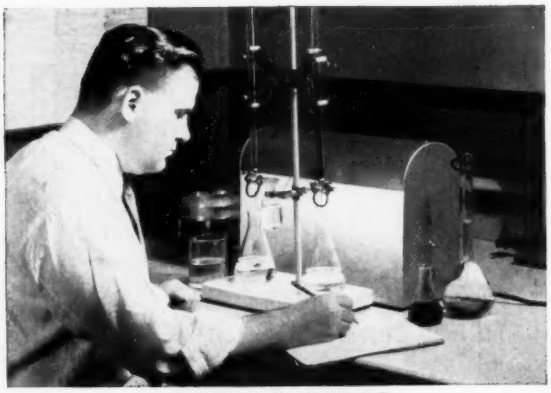

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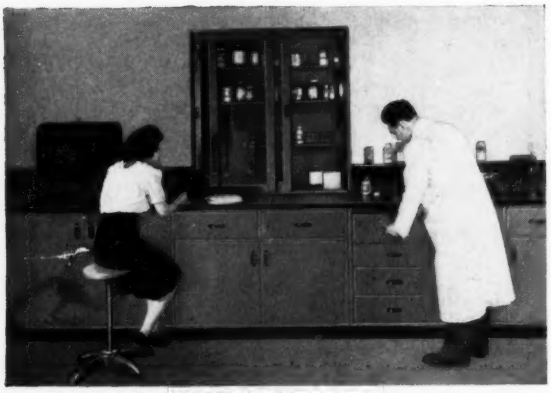

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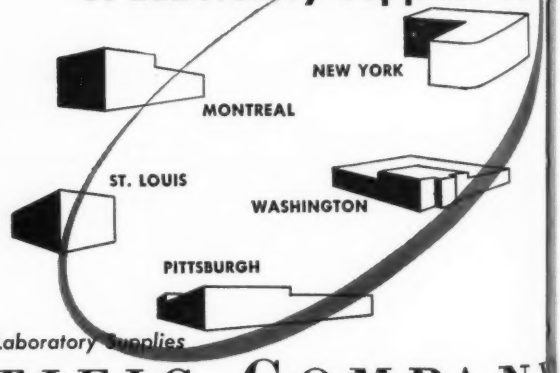

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